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MARTIE E. HAMILTON, B.Sc.

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CONTENTS

PAGE

EXPERIMENTS IN THE USE OF DDT AGAINST THE PASTURE COCKCHAFER, *Aphodius howitti* Hope, by P. B. Carne, B.Agr.Sc. ..

SEASONAL SEVERITY OF THE ATTACK OF THE RED-LEGGED EARTH MITE (*Halotydeus destructor*) ON SUBTERRANEAN CLOVER, by K. R. Norris, M.Sc. 7

STOCK AND SCION INVESTIGATIONS. VI. FURTHER NURSERY TRIALS WITH APPLE ROOTSTOCKS, by L. A. Thomas, M.Sc. 16

THE CATION EXCHANGE CAPACITY OF SOILS AND SOIL COLLOIDS. 1 VARIATION WITH HYDROGEN ION CONCENTRATION, by J. S. Hosking, M.Sc., Ph.D. 21

THE CATION EXCHANGE CAPACITY OF SOILS AND SOIL COLLOIDS. 2. THE CONTRIBUTION FROM THE SAND, SILT, AND CLAY FRACTIONS AND ORGANIC MATTER, by J. S. Hosking, M.Sc., Ph.D. 38

EFFECT OF MECHANICAL COMPOSITION AND NITROGEN CONTENT ON THE WATER-STABLE AGGREGATION OF SEVERAL SOILS, by G. B. Clarke, B.Sc. 51

AN INVESTIGATION INTO THE INHOMOGENEITY OF DEFORMATION IN WIRE DRAWING AND ROLLING, by June Collins, B.Sc. and R. W. K. Honeycombe, M.Sc. 59

Notes

NEW ELECTRICAL AND PHOTOMETRIC UNITS 67

ABSTRACTS OF FORTHCOMING PUBLICATIONS 69

RECENT PUBLICATIONS OF THE COUNCIL 70

FORTHCOMING PUBLICATIONS OF THE COUNCIL 73

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Experiments in the Use of DDT against the Pasture Cockchafer, *Aphodius howitti* Hope

*By P. B. Carne, B.Agr.Sc.**

Summary

The possibility of controlling *A. howitti* infestations in sown pastures at Dickson, A.C.T., was tested using 2 per cent. DDT applied as a dust in talc. Population counts using 180 one-square-link quadrats showed poor control at two months, and almost 100 per cent. control at eleven months after treatment.

The evidence indicates that DDT persisted in the soil for at least six months and killed first instar larvae of the generation following that present at the time of treatment.

1. Introduction

In June 1946, a fairly heavy infestation of the cockchafer, *Aphodius howitti* Hope, a pest of pastures and lawns, was found in sown pastures at the C.S.I.R. Experiment Station, Dickson, A.C.T., a few miles north-east of Canberra, where an ecological study of this and other cockchafers was subsequently initiated. The economic status of the pest has been discussed by Evans (1941), Swan (1934), Twentyman and Pescott (1942).

A. howitti was causing damage to certain experimental plots, so, at the request of the Division of Plant Industry, the writer attempted control of the pest using DDT. The site selected for the test was a half-acre exclosure in a paddock that had been sown in May 1942 with a pasture consisting of *Phalaris tuberosa,* lucerne, and subterranean clover, using superphosphate at the rate of 112 lb. per acre. The soil type present was a greyish-brown clay loam.

The exclosure, known as "P99", was being used for a fertilizer-grazing trial. It was divided into blocks (A–F: see Fig. 1), each block being subdivided into 30 plots, each of 20 by 25 links, which had received various quantities of superphosphate and lime, randomly replicated within each block. The long axis of P99 lies in approximately an E-W direction.

2. Methods

In July 1946, two quadrats, each of 1 link square, were pegged out in the north-eastern corner of each of the 180 plots of P99, each quadrat representing 1/500th of the area of the plot. This location

* An officer of the Division of Economic Entomology.

of the quadrats was dictated by the need to avoid interference with pasture-yield quadrats forming part of the fertilizer-grazing trial. The quadrats enabled two samplings, each of 30 quadrats, to be made of the cockchafer population in each block, one before and one after the DDT treatment.

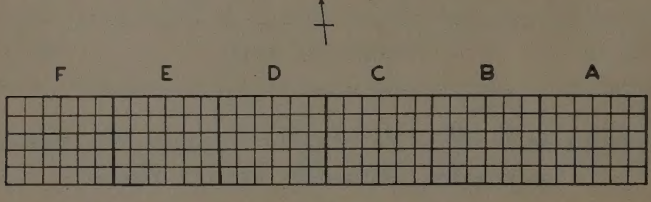

FIG. 1.—Diagram of exclosure P99 showing subdivision into blocks and plots.

The top six inches of soil was dug from each quadrat, and after removal of plant material, was sieved twice, first through a ¼-in. mesh and then through an ⅛-in. mesh, using the apparatus shown in Plate 1, Fig. 1. After washing and fixing, the *Aphodius* larvae were separated from the Melolonthid larvae associated with them, and the two groups counted. The Melolonthids included several species.

Owing to unfavourable weather conditions, application of the insecticide was delayed until August 29. The DDT was used as a dust containing 2 per cent. of the pp-isomer in talc, and was applied by means of a Root barrow-type four-row cropduster, at the rate of 166 lb. per acre. The dust was applied in swathes along the E-W axis of P99 within the blocks A, C, and E, bags being laid over the quadrats of the control blocks B, D, and F, to prevent dust drifting on to them. Care was taken to prevent excessive dusting at turning points, the machine being turned between the rows of quadrats. All the quadrats of each row were dusted in a single run, the overlap of successive swathes falling between the quadrats.

Between October 15 and 29, the second of each pair of quadrats was dug, and the populations again determined.

A rough population survey, based on the presence or absence of casts,* was made of the treated and untreated blocks the following May (1947), to examine the possibility of a residual effect of the DDT. This, of course, dealt with the generation following that of 1946. The results were surprising, and consequently a third population sampling was carried out between July 1 and 23. In each plot, an additional quadrat, adjacent to the original pair, was sampled, using a sieving machine designed by the writer (see Plate 1, Fig. 2).

3. Results

The results of all population counts are shown in Table 1.

While the crude figures for the populations of *Aphodius* at July 1946 and October 1946 suggest a control of approximately 20 per cent.

* Mounds of loose earth thrown up by the larvae in the process of burrow construction.

TABLE 1.—SUMMARY OF RESULTS OF POPULATION COUNTS

—	Block	*Aphodius*: Mean Population per sq. link				Melolonthids: Mean Population per sq. link			
		July 1946	October 1946	G.M.* Survival	July 1947	July 1946	October 1946	G.M.* Survival	July 1947
Treated	A	20·4	15·1	%	0·1	2·6	1·6	%	4·0
	C	5·7	6·0	80·2	nil	2·2	1·5	78.5	4·9
	E	2·8	2·2		nil	2·2	2·3		4·6
	Mean	9·6	7·7		0·03	2·3	1·8		4·5
Control	B	15·2	16·2		8·0	2·6	1·5		4·4
	D	3·9	3·8	103·3	4·9	2·5	1·9	62.5	7·8
	F	3·0	2·7		1·5	2·0	1·0		5·2
	Mean	7·3	7·6		4·8	2·4	1·4		5·8

* The G.M. Survival is the weighted geometric mean of the percentage survival between July and October 1946, the weights being determined by the initial and final numbers in each block.

by the latter date, this reduction cannot be regarded as significant ($P = 0{\cdot}10$); however, there is a chance that it may be real.

The only other factor, apart from natural mortality, which could have contributed to a reduction of the population is emigration. However, field observations suggest that little if any migration occurred on the site, the larvae having remained inactive from before the date of dusting to the day prior to the beginning of the second population count.

The absence of feeding activity over this period was the result of drought conditions, it being characteristic of *A. howitti* that it comes to the surface to feed only when the top soil is moist. The weather data for the months July to October 1946, at Dickson, show that between the date of dusting (August 29), and the day before the beginning of the second population count, the only falls of rain were of 12, 2, and 3 points. Moreover, the data show that the soil must have been dry on August 29, a persistent strong wind having been blowing almost daily since the last appreciable shower (18 points on August 14).

On October 14, calm conditions and a fall of 39 points resulted in a general night emergence of the larvae.

The Melolonthid population (see Table 1) shows a general decrease between the first two counts, which cannot be ascribed to the DDT, the weighted geometric means of survival in the treated and

untreated blocks being 78·5 and 62·5 per cent. respectively, i.e. survival appears to be highest in the treated areas; however, the treatment difference is not significant ($P = 0{\cdot}15$).

When the populations of *Aphodius* 2 and 11 months after application of the DDT are compared (see Table 1), a highly significant reduction is apparent in the treated blocks A, C, and E, while the population in the control blocks B, D, F, remains at a moderately high level. *Aphodius* larvae were found only in three adjacent quadrats out of the 90 quadrats of the treated blocks, and it is considered highly probable that even these did not hatch in them, but were immigrants from an adjacent untreated area. The quadrats concerned were within twelve inches of the margin of the dusted area, in an outside row, and adjacent to the edge of an advancing front of high density outside the exclosure.

Thus although the DDT had little effect on the almost mature larvae of the 1946 generation,* it apparently persisted on or in the soil, and in some way prevented reinfestation during 1947. The Melolonthid population in July 1947 was much higher than at either of the two earlier counts. Although it was somewhat higher in the control than in the treated blocks, the difference is not significant and no effect of the DDT can be inferred.

4. Discussion

No direct evidence was obtained to explain how the DDT acted in controlling the 1947 infestation in the treated blocks. Thus it is necessary to consider possible ways in the light of circumstantial evidence obtained in the course of the work. The first important fact to consider is that remains of completely hardened *Aphodius* adults were found during the third population count in approximately equal numbers in all blocks. There is little doubt that these are the remains of beetles that had swarmed and oviposited, which are characteristically found at or near the surface and very rarely at the depth at which oviposition takes place. Had they been the remains of adults that had died before swarming, the writer's observations indicate that they would have occurred mostly 4-5 inches below the surface, and would have shown evidence of incomplete melanization and imperfectly developed elytra.

It seems safe to assume, therefore, that *Aphodius* entered the soil in both treated and untreated blocks alike for laying. If this is so, the DDT must have inhibited oviposition, destroyed the eggs, or killed the young larvae by contact with, or after ingestion of soil, when they first burrowed to the surface in April 1947.

The first two possibilities seem most unlikely, as several species of Melolonthids, adults of which swarmed about the same time as *Aphodius,* produced many offspring in both treated and untreated blocks (see Table 1), showing that they were able to oviposit, and that many of their eggs survived. This suggests the third possibility as the correct explanation. This interpretation is supported by the

* A similar dusting of a heavily infested plot in August 1947 gave a parallel result, few larvae being killed, to judge from the continued reappearance of casts on the treated areas.

recent work of Fleming (1947) on *Popillia japonica* Newm. He found that adult *Popillia* burrow into, and oviposit normally in, soils dusted with up to 50 lb. per acre of technical DDT,* and that the eggs hatch normally. He concluded that the larval stage was the only susceptible one, young larvae being the most readily destroyed.

The larvae of *P. japonica* resemble the Melolonthids present in the Dickson pasture, and differ from *A. howitti,* in that they do not come to the surface to feed, but remain in the soil moving about horizontally. To destroy the *Popillia* larvae, the DDT must have been washed down to the level at which they occurred. It is not surprising that a heavy dosage of DDT was necessary (30-35 lb. per acre as against 3·3 lb. per acre at Dickson). Fleming found that the DDT reached the larvae in sandy soils more rapidly than in loams. As has already been mentioned, the Melolonthids on P99 were not affected by the DDT. This is probably explained by the facts that the dosage was light, and that the soil was a rather heavy clay loam—much less permeable than those studied by Fleming.

As hatching of *Aphodius* over the general area of the Experiment Station did not begin until the end of February 1947, and the larvae did not burrow to the surface until mid April, it appears that the DDT must have persisted in the soil for at least six, probably eight, months, in order to give the results obtained. As far as is known to the writer, a persistence of this duration of DDT in the field has not been recorded before, although Fleming reports persistence in excess of 3½ years under laboratory conditions. Observations will be continued in 1948 to ascertain if the populations of *Aphodius* in the blocks treated and untreated in 1946 continue to show significant differences.

The degree of control obtained was very high (virtually 100 per cent.). It is thought probable that the dosage of DDT applied (166 lb. of 2 per cent. dust per acre) is well in excess of that necessary to provide a satisfactory control for practical purposes, as field observations suggest that damage caused by *Aphodius* is negligible below a density of 20-25 per square foot, and really serious only when in excess of 150-200 per square foot.

Thus it may be found that this insecticide can be used economically for the control of *A. howitti,* especially in such restricted sites as sheep camps, holding paddocks, and the like, where infestations are usually most severe. The persistence of the DDT would permit application of the dust at a time suitable to the landholder up to six months before the date of oviposition; indeed it is quite possible that an area, once dusted, may remain immune to economic infestation for longer than the following year.

5. Acknowledgments

The writer wishes to thank Dr. K. H. L. Key of the Division of Economic Entomology for his interest in the present investigation and his helpful suggestions, and Mr. L. R. Clark of the same Division for his criticism of the rough draft of this paper. Acknowledgments

* The pp-isomer content of the technical DDT is not stated, but would probably lie between 60 and 70 per cent.

are due to Mr. G. A. MacIntyre and Miss L. McGovern of the Section of Mathematical Statistics for the analysis of the results, to Mr. W. J. Rafferty who constructed the sieving machine, and to Mr. T. Pickard for the two plates.

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Seasonal Severity of the Attack of the Red-legged Earth Mite (*Halotydeus destructor*) on Subterranean Clover

*By K. R. Norris, M.Sc.**

Summary

Plots of subterranean clover were treated at monthly intervals throughout the 1941 season for the control of the mite *Halotydeus destructor*. The yield of plots protected from the time of germination was superior to that of all other treatments. Plots treated in mid-May, about two weeks after germination, produced yields of subterranean clover intermediate between those treated from the time of germination, and those treated from mid-June onwards. By mid-June the plants could no longer benefit from the destruction of the mites. Possibly by this time the competition of other plant species was too severe to permit the recovery of the stunted subterranean clover. The yields of subterranean clover seed parallel those of the foliage.

Other plant species were little affected by the variation in mite attack.

No yield difference resulted if the acaricidal treatment was applied repeatedly instead of once only.

The significance of the results is that pastures must be protected from the time of germination. The possibility of achieving this result with DDT is indicated.

1. Introduction

The importance of the red-legged earth mite *Halotydeus destructor* (Tucker) (Acarina, fam. Penthaleidae) in relation to subterranean clover pastures in Western Australia was discussed by the author in a previous publication (1944), in which, however, it was not possible to indicate the time of year at which most critical damage is done by the earth mite. The experiment described in the present paper was designed to investigate this point more exactly.

When this investigation was undertaken, the toxicants available were unsuitable and uneconomical for the control of the earth mite in pastures, but the results obtained assume importance in the light of the discovery of the toxicity of DDT to the earth mite.

2. Design of the Experiment

It was decided to compare the yields of plots treated with an acaricide at approximately the mid-date of each month of the season during which the earth mite is active at Katanning, Western Australia. Provision had therefore to be made for mid-monthly treatments in May–November inclusive, this representing approximately the limits of the mite season in the district. Plots were also included which were to be kept free of mites from the beginning to the end of the season, and others on which the mites were to be unchecked from the beginning to the end of the season. It was originally intended to carry the observations on long enough to find whether the late 1941 treatments reduced the numbers of aestivating eggs matured, and so

* An officer of the Division of Economic Entomology.

reduced the chances of damage to the seedlings in the 1942 autumn, but war-time conditions prevented the continuance of the work after 1941.

The acaricidal treatment adopted was a bait consisting of a solution of sodium fluoride and cane sugar absorbed on cereal chaff (Norris 1943). Eggs are unaffected by this treatment, and usually some at least hatch and give rise to surviving mites after it has been rendered ineffective by rain. In addition, mites may remain inactive for several days during ecdysis, and in this way escape a treatment of short persistency. The effectiveness of a non-persistent toxicant may thus vary with the stage of development of the mite population. It is also influenced by rain, and so may vary greatly from time to time during a season. To obtain information on this point, two series of plots were incorporated in the experiment. In one series the mite population was kept as low as possible by repeating the treatment whenever necessary; in the other series only one treatment was applied, and any surviving mites were left without further interference for the remainder of the season. Eighteen treatments were thus employed, as detailed in Table 1.

TABLE 1.—DETAILS OF TREATMENTS*

A. Repeated Treatment to keep Mite Population as Low as Possible after Initial Treatment.	B. One Treatment Only
1. Free of mites entire season	1. Free of mites entire season
2. Initial treatment mid-May (13)	2. Treated mid-May (13)
3. Initial treatment mid-June (13)	3. Treated mid-June (13)
4. Initial treatment mid-July (14)	4. Treated mid-July (14)
5. Initial treatment mid-August (14)	5. Treated mid-August (14)
6. Initial treatment mid-September (15)	6. Treated mid-September (15)
7. Initial treatment mid-October (14)	7. Treated mid-October (14)
8. Initial treatment mid-November	8. Treated mid-November
9. Mites uncontrolled entire season	9. Mites uncontrolled entire season

* Treatment 1A was identical with 1B, and 9A identical with 9B. Numbers in brackets indicate actual dates of the application or initial application of the treatments. The mites died out before the mid-November treatment could be applied. Thus treatment 8 of each series was comparable in every respect with treatment 9.

Four replications were laid out in a split-plot randomized block, the plots being in eight rows of nine. Each plot was 7 links square, and accessible from all sides from pathways.

3. Establishment and Maintenance of Plots

The experiment was carried out on "Glenalbyn", the property of Messrs. Ball Bros., a few miles south-west of the township of Katanning. The soil at the site of the plots was a grey, sandy loam on which oats had been grown in 1940. The selected area was sown in the third week in April 1941 with First Early subterranean clover seed which was of high purity and germination and had been inoculated with suitable *Rhizobium* culture the day before sowing. The site was first raked lightly to remove trash and to scratch up the surface. After the even hand-distribution of the seed (12 lb. per acre) and superphosphate (1½ cwt. per acre) more vigorous raking was performed to bury the seed. Seedlings from unburied seeds suffered some mortality from drought and, to make up for this loss, seed equivalent to about 2 lb. per acre was sown on each plot on May 9.

Each plot was fenced with a 6-in. strip of sheet metal embedded 2 inches into the ground. These barriers were painted frequently with creosote, the repellency of which ensured that the mite population of each plot remained as a unit.

At the appropriate treatment times 2 oz. of the wet bait mixture were sprinkled on each plot to be treated. (After the commencement of the experiment sodium arsenate was found to be a more satisfactory poison, but the use of sodium fluoride was continued for the sake of uniformity.) In case the addition of sugar and chaff to some of the plots and not to others should have produced any differences in soil fertility, a dressing of the unpoisoned mixture was applied twice during the season to plots on which the poison bait had not been spread. No pests other than the red-legged earth mite appeared on the plots in numbers likely to have affected the results of the experiment. The plots were securely fenced against stock and rabbits and were not grazed at any time.

Rainfall and temperatures during the period of the experiment were not in any way abnormal (Norris 1944, Table 3, p. 16).

4. Mite Population

Several small mites were found in locally favourable places near the plots on April 25. By April 29 general hatching had occurred over the area of the plots. Small capeweed seedlings were present on the land a week or so before this date and served as food for the young mites on emergence. Subterranean clover germination commenced by April 29, and within a few days slight mite-damage could be seen on some of the seedlings.

The high population of mites hatching from the aestivating eggs was responsible for severe damage to subterranean clover seedlings observed on untreated plots in the second week in May. Early in June the population on untreated plots had dwindled considerably. By the first week in July it was evident that the second generation was developing, as there were a few large mites and very large numbers of small mites. From this time the mite population on untreated plots remained fairly high until about the middle of October when the numbers began to decline. By the end of October very few mites were present, and none at all could be found after the first week in November.

On repeatedly-treated plots a high degree of control was obtained, and in the few cases where more than average numbers persisted the mites were still not numerous enough to inflict any visible damage on new growth on the plants. On plots treated once only, the kill was invariably good, but in less than two months the multiplication of the survivors often produced a population at least equal to that on untreated plots.

5. Growth and Yield of Plots

Subterranean clover (*Trifolium subterraneum* L.) was the only species sown on the plots. The only important volunteer species was capeweed (*Cryptostemma calendulaceum* R.Br.). *Erodium botrys* Bertol., minor clovers, oats, and annual grasses occurred very sparsely.

Two samples, each 1 link square, were taken from each plot on October 13. All foliage on each sample area was cut to ground level with a knife. After air-drying the samples were sorted into three fractions: subterranean clover, capeweed, and other species. The fractions were oven-dried before weighing. Apart from the results of this sampling, only general notes on the growth of the plots can be presented.

On each plot two areas of 1 square link were sampled for subterranean clover burrs after the plants had completely dried. The seed was hand-separated and weighed.

(*a*) *Subterranean Clover* (*Trifolium subterraneum* L.)

Germination of the subterranean clover commenced by April 29, a week or so later than the earliest germination noticed in the pastures on the farm, but close enough to the general germination for the clover of the experimental plots to be comparable with self-sown plants.

Very slight damage was suffered by the seedlings in the 1A and 1B treatments before it was possible to eliminate the first-hatched mites. By May 14, except on treatments 1A and 1B, many seedlings were severely damaged. By May 22 the heavily damaged seedlings of treatments 2–9 of both series were also suffering severely from drought, only 3 points of rain having been recorded since May 6. It was thought that many seedlings were dead, but falls totalling 24 points recorded on May 29–30 were adequate to save most of them, and new leaves were being put out by June 3, in spite of the complete withering of the cotyledons. Seedling mortality observed on the plots was very low. Subterranean clover seedlings in pastures on the farm were observed to experience a history of mite and drought damage similar to that observed on these plots.

The subterranean clover on plots 1A and 1B, and on plots 2A and 2B (which had received adequate rain up to May 13, the date of destruction of the mites) was not obviously affected by the drought.

New germination was noticed as late as June 13. On plots to which the acaricide had not been applied, such late seedlings were very severely damaged.

On July 11 the subterranean clover on 1A and 1B plots was uniformly well-grown and undamaged. That on 2A and 2B plots, which had been treated on May 13, was undamaged and showed fair

growth. On the remainder of the plots, including 3A and 3B treatments (treated June 13) most of the clover was stunted and severely damaged.

On August 18 little change in the relative growth of the plots was noticeable. No damage was detected on 1A, 1B, 2A, and 3A plots. On the 2B and 3B plots, where the mites had been allowed to multiply after treatment, a fair amount of damage was detectable, though the growth was not less than on the corresponding plots of the A series. On the remainder of the plots there was a fair amount of damage, though generally less than recorded in July.

Much the same conditions were found on September 15. At this stage, however, although the clover on the 2B and 3B plots was vigorous and comparable in growth with that on the 2A and 3A plots respectively, it showed quite extensive mite-damage. Further detailed notes made on October 11 showed much the same conditions as in September. The clover on treatments 2B and 3B was fairly severely damaged. There were no obvious differences in clover growth between treatments 3–9 in both A and B series.

Much of the subterranean clover had wilted by the end of October and it was all dead by the end of November.

The weights of the cuttings made on October 13 are presented in Table 2. The importance of the attack of the earth mite on the seedling stage is clearly demonstrated by these figures. Even on treatment 2A, from which the mites were absent except for the fortnight following germination, the clover yield was inferior to that of treatment 1A, which was free of mite attack from the time of germination. It is significant, too, that the yield of treatment 2B was not less than that of 2A, although by September the mites surviving the treatment of May 13 on 2B had multiplied to numbers comparable with, if not greater than those on untreated plots. It is reasonable to conclude that all important mite-damage to subterranean clover occurred in the first few weeks after germination.

The yields of subterranean clover seed (Table 3) closely parallel those of the foliage.

(*b*) *Capeweed* (*Cryptostemma calendulaceum* R.Br.)

In 1940 a vigorous growth of this species was present in the oats on the site of the experiment. There was a fair number of young seedlings present on the plots when the clover was sown in the third week in April 1941, and most of them were little affected by the sowing. By May 22 the capeweed growth was so vigorous that it was considered advisable to trim some of the denser clumps with shears. The greater part of most of the plots was untouched by this treatment, and the short growth of subterranean clover was completely untouched. The capeweed flourished until late September, after which its vigour declined.

The mites fed freely on this plant, but no suggestion was ever obtained that its growth was affected by the attack. This is borne out by the yield figures (Table 2), which show no differences which can be considered significant.

TABLE 2.—YIELDS IN CWT. PER ACRE FROM CUTTINGS MADE ON OCTOBER 13, 1941.*

Treatment	Subterranean Clover			Capeweed			Other Species			Total Foliage Yields		
	A	B	Mean	A	B	Mean	A	B	Mean	A	B	Mean
1. Mite-free all season ..	10·16	9·51	9·83	20·64	21·40	21·02	6·12	6·52	6·32	36·92	37·44	37·18
2. Treated May 13 ..	5·30	6·62	5·96	27·99	21·25	24·62	2·00	5·32	3·66	35·29	33·20	34·25
3. Treated June 13 ..	2·44	0·92	1·68	24·45	25·17	24·81	3·23	2·24	2·73	30·12	28·33	29·23
4. Treated July 14 ..	1·79	1·65	1·72	21·96	20·12	21·04	2·27	2·96	2·61	26·01	24·74	25·36
5. Treated August 14 ..	1·11	1·44	1·28	20·89	20·52	20·70	3·23	3·95	3·59	25·23	25·91	25·59
6. Treated September 15 ..	1·84	0·75	1·29	25·54	23·58	24·56	2·77	5·04	3·90	30·16	29·38	29·76
Mean 7 + 8 + 9, untreated..	1·46	1·46	1·46	23·45	24·25	23·85	4·33	3·44	3·88	29·24	29·14	29·19
Critical differences 5%	3·62	3·62	2·56	7·38	7·38	5·22	3·79	3·79	2·68	6·34	6·34	5·73
Critical differences 1%	4·83	4·83	3·42	9·84	9·84	6·96	5·06	5·06	3·58	8·45	8·45	7·79

* In Series A the plots were re-treated as often as necessary for mite-control after the date of initial treatment indicated in column 1. In Series B the plots other than 1B were treated once only on the date indicated. Treatments 7 and 8 had not been applied when the cuttings were made, and so for consideration in this table, treatments 7-9 are identical. The yields have therefore been averaged. Slightly greater precision is possible in the comparison of these pooled figures with other treatments, but no fresh significant differences are introduced.

TABLE 3.—YIELD OF SUBTERRANEAN CLOVER SEED (CWT. PER ACRE)*

Treatment	Series		Mean A + B
	A	B	
1. Mite-free all season	4·04	2·99	3·51
2. Treated May 13	3·09	1·80	2·44
3. Treated June 13	0·37	0·82	0·59
4. Treated July 14	1·17	0·42	0·79
5. Treated August 14	0·66	1·03	0·84
6. Treated September 15	1·31	0·50	0·90
7. Treated October 14	0·30	0·19	0·24
Mean 8 + 9, untreated	0·60	0·40	0·50
Critical differences 5%	1·40	1·40	0·99
Critical differences 1%	1·87	1·87	1·33

* The clover and mites died before it was possible to apply treatment 8, which has therefore been pooled with treatment 9, with which it is identical.

(*c*) *Other Species*

This fraction of the foliage samples contained species too poorly represented on the plots for separate assessment of yield. The principal elements were *Erodium botrys*, minor clovers (e.g. *Trifolium procumbens* L., *T. glomeratum* L.), self-sown oats and various annual grasses, such as silver grass (*Vulpia* spp.). No consistent trends are shown by the yield figures (Table 2), except that the high yield of both 1A and 1B treatments suggests that some of the constituents were either susceptible to mite attack, or particularly sensitive to association with subterranean clover. Generally, however, subterranean clover has but a slight influence on soil fertility in its first year of growth on ungrazed land.

(*d*) *Total Yields*

The yield of treatment 1 in each series was significantly superior to that of the remaining treatments, except treatment 2, which was intermediate between 1 and the remainder of the treatments (Table 2). The yield of subterranean clover was the dominant influence in the production of this effect.

6. Discussion

This experiment showed clearly that the greatest check to the growth of the subterranean clover was inflicted by the mites during the first few weeks after germination. The occurrence of a practically rainless period from May 6–29 probably considerably accentuated the effect of earth mite attack over the period, although some damage occurred before this date when rainfall was adequate

for clover growth. Such long drought periods following the germination of subterranean clover are not an invariable feature of the Western Australian climate, but occur frequently enough in the drier areas of the subterranean clover belt to be a serious hindrance to the establishment and maintenance of the pastures (Norris 1944; Meadly 1946). Unseasonable germinations as early as that recorded by Meadly (loc. cit.) at Kojonup in 1942 (March rains) undoubtedly result in the death of subterranean clover seedlings from drought alone, as temperatures do not permit the hatching of earth mite eggs before late April or early May. However, it is clear that the importance of rainless periods occurring after the normal hatching time of the mites would be lessened if the mite could be brought under control.

By mid-June the subterranean clover plants no longer benefited from the destruction of the mites. Probably this was due to their domination by capeweed at this stage. The subterranean clover was able to compete successfully with this species if freed from mite attack early in the season.

The significance of the results of this experiment to the practical problem of mite control lies in their clear indication that the pest must be attacked as soon as possible after hatching in the autumn. At this time the clover plants are in the greatest need of protection: the mites are also exceedingly delicate, and have laid no eggs on the herbage. To achieve such early control with non-persistent acaricides the time of application must be determined by careful observation, a matter of difficulty to the farmer on account of the minute size of the young mites. The cost of non-persistent acaricides of known effectiveness, and the labour charges involved in their distribution, exceed the maximum annual return from sheep pastures in Western Australia. Furthermore, the mite population has such great powers of recuperation after reduction by non-persistent toxicants that the expense would be at least annual if control were to be maintained.

The recognition of the toxicity of DDT to the earth mite (Kemp 1946; Dumbleton 1947) in conjunction with its well-known quality of great persistency opens up a new avenue in the investigation of the control of the mite. Persistency for little more than a week would deprive the mite population of its recuperative powers, as there would still be a lethal concentration of the acaricide after the hatching of all the eggs, and the resumption of activity by mites that were moulting at the time of application. If it should prove possible to apply the toxicant before the hatching of the mites, concern over the critical time for treatment in the autumn would be obviated. A mechanism for application is already in existence in the routine top-dressing of pastures with superphosphate. This fertilizer is generally applied in advance of the autumn rains, when pressure of other farm work is light. Should DDT prove persistent enough, its incorporation in the fertilizer may be possible, thus economizing on labour and equipment for distribution.

7. Acknowledgments

Thanks are tendered to Messrs. Ball Bros. for permission to carry out the work on their property. Mr. A. T. Mills assisted with the establishment and maintenance of the plots, and the sorting of the herbage samples. Mr. G. A. McIntyre assisted with the plans of the experiment, and performed the statistical analyses of the results.

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Stock and Scion Investigations

VI. Further Nursery Trials with Apple Rootstocks

*By L. A. Thomas, M.Sc.**

Summary

In the search for apple rootstocks superior to Northern Spy, both local and imported stocks reputedly immune to woolly aphis have been tested with Jonathan as the scion variety. Two techniques were used in establishing the trials: one, by budding the stocks while still growing in the layerbed and then transplanting into permanent positions in the dormant season; the other, by planting the stocks in permanent positions and budding them the following summer.

Woolly aphis attacked reputedly resistant stocks. Merton 778 and local selection S4 appear to be desirable improved rootstocks.

1. Introduction

The two nursery rootstock trials described herein as Trial B and Trial C are briefly referred to in a previous publication (Dickson and Thomas 1938). The rootstocks in Trial B consist of two local selections together with Northern Spy, while those used in Trial C have been reported by various authors to be resistant to attack by woolly aphis. These stocks were tested with the object of finding a more suitable stock for the district than the commonly used Northern Spy.

2. Material and Methods

Trial B

In this trial, Northern Spy was compared with a local selection K, which was obtained from a vigorous tree in a planting of Jonathan on seedling stocks of unknown origin, and with S4, another local selection which has been accepted by orchardists as Northern Spy. In nine orchards where S4 has been recognized, the scions are either Jonathan or Granny Smith, and the trees are all outstanding and have a more upright growth habit than trees on Northern Spy.

These stocks were budded to Jonathan while still growing in stoolbeds in February 1936, and were cut off and planted in randomized blocks to form this trial in July 1936. This method of establishment was adopted in order to save a year in commencing the trial: it dispensed with the usual operation of planting rootstocks in rows for budding in the succeeding summer. The trees were spaced 3½ feet apart in rows 5 feet distant, with six replications of four-tree units.

Trial C

The apple varieties Lady Carrington, Ben Davis, Allsopp's Early, Niedwetzkyana and Zuccamaglio were obtained from East Malling as maiden trees, and were planted to form layerbeds and propagated in this manner to obtain the varieties on their own roots. One-year-old rooted shoots (stocks), together with similar material of the two Merton stocks, Nos. 778 and 779, and Northern Spy were planted in

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randomized blocks in July 1936 and budded to Jonathan in February, 1937. In this trial the planting distance was 2 feet apart in rows 5 feet distant, with 40 trees on each rootstock. It was intended to remove alternate trees when tree growth decreased, but this was done in 1940, at an earlier date than anticipated owing to the vigorous tree growth. Up to the time of removing these trees no fruit had matured.

In both trials a similar method of training the trees with a central leader was adopted, severity and type of pruning were similar, the same amount of fertilizer was applied, and cultivation was uniform. The annual extension growth was measured in cm. until lack of labour during the war period made this an impossible task; the weight in lb. of the annual crops borne from 1941 to 1944 was recorded, and finally, the weight in lb. of the scions above the union was obtained in 1944.

3. Results

Trial B

In the nursery the stocks were produced from stoolbeds and observations were made over a period of three years. Stock K is clean-stemmed, of medium growth, and like Jonathan in appearance; it is somewhat thin-barked for budding but roots fairly readily, with thin, hard roots. It proved to be only moderately resistant to woolly aphis attack. Stock S4 is a tall clean-stemmed stock, vigorous in growth and fairly resistant to woolly aphis. It roots readily with abundant short fibrous roots and transplants easily.

A statistical analysis was made of the annual growth measurements for the year 1941, of the total growth for the years 1937 to 1941, and of scion weight in 1941. The results of these analyses are summarized in Table 1.

TABLE 1.—ANALYSIS OF THE RESULTS OF TRIAL B

Stock	Mean Growth Increments per Till		Mean Till Weight in 1944
	Growth 1941	Growth 1937–38–39–40–41	
	cm.	cm.	oz.
Spy	968	4,788	69·29
K	1,470	6,243	99·74
S4	1,508	6,705	136·27
Minimum difference for significance :—			
At 5 per cent	304	1,048	22·91
At 1 per cent.	450	1,554	33·98

The variation in tree size observed in this experiment is thought to be largely the result of the method of establishing the trials. Factors contributing to this variability are (*a*) the differences in the amount of roots produced on one-year shoots from layerbeds, and (*b*) the varying degree of successful establishment following transplanting.

Further, in normal planting practice, a selection is made of the better-grown trees for planting into permanent positions, but under the conditions of this trial no selection was possible.

TABLE 2.—AVERAGE ANNUAL CROPS (LB. PER TREE)

Stock	1941	1942	1943	1944	Total
Spy	8·2	12·3	6·9	11·2	38·6
K	7·3	15·4	8·9	13·6	45·2
S4	1·9	10·8	5·9	11·8	30·4

The data concerning crops borne are given in Table 2. The average weight for a single fruit over the whole cropping period is 4·1 oz. from trees on Stock K, 4·0 oz. for those on stock S4, and 3·5 oz. from trees on Spy stocks. These results are interpreted to mean that under the competitive conditions of close planting, the fruit from trees on Spy stocks were smallest, but it is thought that under orchard conditions with trees spaced at 20 feet or more apart the differences would not be maintained, except during extreme drought.

In short-term nursery stock trials the cropping figures can only disclose tendencies in cropping habits. In this case it appears that Spy has again shown ability to produce early and heavy crops, and that Stock K appears to have the same habit. Stock S4, which has produced the largest trees, is slower in coming into cropping, but by 1944 had caught up with Spy. From observations made in orchards where trees on Spy and S4 occur together, the indications are that trees on S4 will produce larger crops than those on Spy.

Trial C

A statistical analysis was made of the growth in 1940 and of the total growth 1938 to 1940. This information is summarized in Table 3 together with other pertinent data.

TABLE 3.—ANALYSIS OF THE RESULTS OF TRIAL C

Stock	Mean Growth Increments per Tree		Average Weight of Trees as Percentages, 1944 Northern Spy = 100	Average Crop per Tree 1941–1944
	Growth 1940	Total Growth 1938–1940		
	cm.	cm.		lb.
Merton 778 ..	1,966	4,042	266·5	32·2
Zuccamaglio ..	1,816	3,940	246·9	31·7
Ben Davis ..	1,888	3,848	255·8	14·1
Allsopp's	1,658	3,612	198·8	37·5
Merton 779 ..	1,698	3,478	196·9	40·5
Lady Carrington ..	1,548	3,456	149·2	33·9
Northern Spy ..	1,252	2,608	100·0	20·8
Niedwetzkyana ..	896	2,098	104·5	15·3
Minimum difference for significance :—				
At 5 per cent.	270	549	..	..
At 1 per cent.	361	732	..	..

Other characters of these rootstocks are summarized below:—

Merton 778. A stock immune to woolly aphis, with coarse roots, produces an even and deeply penetrating root system, propagates readily.

Merton 779. Immune to woolly aphis; roots readily in stool-beds with abundant short fibrous roots; produces an uneven and fairly shallow root system; the stock stem is buttressed, showing a trefoil or quatrefoil shape in cross section.

Zuccamaglio. This stock has proved to be very susceptible to woolly aphis.

Ben Davis. Susceptible to woolly aphis; trees on this stock have grown well, but fruited poorly.

Allsopp's. Fairly resistant to woolly aphis attack; roots are thin but deeply penetrating.

Lady Carrington. Produces a shallow-rooted stock which commences growth early in spring; but the stock and trees budded on it are subject to frost damage, locally called " sour-sap ".

Niedwetzkyana. Has proved susceptible to woolly aphis; Jonathan worked on it makes a small unthrifty tree.

Resistance to woolly aphis attack.—At the time that this trial was planted, it was known that some stocks, e.g. Ben Davis, were susceptible to attack by woolly aphis in Australia, but as these varieties were reported to be resistant in Europe, it was thought that varietal strains more resistant to woolly aphis attack might exist. However, it was soon evident in the nursery that this imported material was susceptible, and our experience is in agreement with that of Greenslade (1936) who quotes examples of apple varieties resistant to woolly aphis in some fruit-growing areas and proving susceptible in others.

Shoot:stem ratios.—As some of these rootstocks, viz. Lady Carrington, Zuccamaglio, and Allsopp's, have been grown as own-rooted trees (Thomas 1944), and the ratio of shoot weight to root weight determined, it was decided to test the hypothesis that the S/R ratio determined from own-rooted trees, remained constant when these varieties were used as rootstocks with a different scion variety.

Accordingly, four Jonathan trees on Allsopp's and four on Lady Carrington rootstocks were excavated in the dormant season and the weight of shoots and roots obtained. From the previous trial (Thomas 1944), the S/R ratios for own-rooted trees of Allsopp's and Lady Carrington were found to range from 4·0 to 4·4 and from 2·2 to 2·5 respectively; with Jonathan as a scion on these varieties, the ratios determined were for Allsopp's from 0·8 to 1·1, and for Lady Carrington, from 0·9 to 1·1. These values appear to bear no relationship to those obtained from own-rooted trees, or to those of own-rooted Jonathan trees with values ranging from 2·1 to 2·5 (see Thomas 1944).

It was noted, however, that the rooting habit of these two varieties was essentially the same, whether grown as own-rooted trees or with a Jonathan scion.

4. Acknowledgment

The writer gratefully acknowledges the assistance of Mr. G. A. McIntyre, Section of Mathematical Statistics, who made the statistical analyses of the data.

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The Cation Exchange Capacity of Soils and Soil Colloids
1. Variation with Hydrogen Ion Concentration

*By J. S. Hosking, M.Sc., Ph.D.**

Summary

A rapid method for determining the cation exchange capacities of clay minerals, soils, and soil colloids, and their variations with hydrogen ion concentrations, is described; it involves the leaching of samples as small as 0·1g. with ammonium acetate solutions adjusted to pH values from 5 to 10, and the subsequent estimation of the ammonium absorbed.

The results have been investigated for a series of soils including types ranging from podsols to chestnut earths, for colloids separated therefrom, and for samples of the clay minerals montmorillonite, halloysite, and kaolinite, characteristic of these soils. For all the samples the cation exchange capacities are shown to increase from a minimum value at pH 5 to a maximum value at pH 9 with a subsequent sharp decline with further increase in the pH. The rate of increase in the cation exchange capacity per unit pH varies, for samples examined, from 0·5 to 3·0 m.e. per 100 g. and this variation is independent of the clay mineral type, of the soil type, and of the magnitude of the cation exchange.

The maximum cation exchange capacities at pH 9 varied from 5 to over 100 m.e. per 100 g., and in general those soils or colloids characterized by the montmorillonite type mineral showed the highest values. Certain soils, however, in which clay minerals of the kaolin type alone have been identified, gave much higher capacities than expected. In some instances the results may be related to the probable presence of the mineral halloysite; in others amorphous clay materials with high exchange capacities are undoubtedly present. It is considered that such amorphous exchange materials play a far more important role in soils than has been attributed to them in recent years.

In view of the practical significance of the cation exchange capacity at the pH value of the soil itself, it is considered that the term "cation exchange capacity of the soil" should be used for the value at this pH and that the term "maximum cation exchange capacity" should be used for the value at pH 9; for the comparison of the exchange capacities of soils and colloids the maximum value should be used.

For complete characterization of the cation exchange reactions of the soil, the cation exchange capacity and the maximum exchange capacity should be given, and in addition the magnitude of the increase or decrease per pH unit with change in pH from that of the soil, should also be stated.

1. Introduction

The materials, such as clay minerals and complex organic compounds, in soils capable of exhibiting the property of cation exchange, are in reality weak multibasic acids in which the hydrogen ions are distributed over the exposed surfaces of large insoluble anions. These hydrogen ions may be wholly or in part replaced by metallic cations. Since these exchange materials are weak acids, complete neutralization of the hydrogen ions capable of dissociation will not take place at the neutral point, pH 7, but at a more alkaline reaction, the equivalence point. In practice, the equivalence point of the clay minerals and

* An officer of the Building Materials Research Laboratory. The work described was carried out in the laboratories of the Council's Division of Soils and of the Division of Plant Nutrition, University of California, while the author was an officer of the Division of Soils.

of the organic acids as they occur admixed in soils is extremely difficult, if not impossible, to determine; as a result the concept of saturation (Ramann 1911; Hissink 1924) has been introduced, and various reaction values have been adopted as standard reference points at which to express saturation or cation exchange capacity.

Of these values two, pH 7 and pH 8·4, have a special though not necessarily a fundamental significance. The neutral point, pH 7, is of agronomic importance and the determination of the capacity at this value is based upon a desire for information concerning the lime required to neutralize an acid soil; pH 8·4 is of pedological importance since free calcium carbonate, indicative of the change-over from pedalfers to pedocals, may appear in the soil at this pH value.

The methods employed for the determination of the capacity are many and varied. The most general procedures embrace the leaching of the sample with a buffered salt solution, and either the estimation of the total amount of the cations absorbed by the soil or, alternatively, the estimation of the individual exchangeable cations in the leachate (hydrogen may be determined separately), and their subsequent summation. Other methods involve the titration with standard alkali of either a suspension of the electrodialysed or hydrogen-saturated sample, or the acid extract obtained by leaching such a sample with a buffered salt solution.

Subsequent to the definition by Gedroiz (1924) of a saturated soil as one which gives a neutral aqueous suspension, or a neutral extract, on treatment with a neutral salt solution, the majority of American workers adopted pH 7 as the standard of reference. The exchange capacity is usually determined by leaching the soil with either neutral normal ammonium or barium acetate and estimating the cations absorbed therefrom by the soil; it is sometimes determined, on the other hand, by the summation of the exchangeable metallic cations, determined in the leachate, and the exchangeable hydrogen obtained by titrating the leachate back to neutrality. Bradfield and Allison (1933) proposed pH 8·4 as a more logical reference point. They based their proposal upon the observation that in most soils of the humid regions calcium is the dominant exchangeable cation, and that in such soils it might be expected that the pH value would not normally rise appreciably above 8·4 (the pH of calcium carbonate in equilibrium with carbon dioxide), since any metallic cations in excess of the amount necessary to neutralize the exchange complex to this value would appear as calcium carbonate. This standard was later adopted by Piper (1936) who estimated the cation exchange capacity by adding the total of the exchangeable metallic cations, determined in an ammonium or sodium chloride extract from soils, to the exchangeable hydrogen determined by a titration procedure embracing the use of *m*-nitrophenol.

However, the reaction value rises appreciably above pH 8·4 in soils containing either magnesium or sodium as the dominant or an important exchange cation; this is quite normal not only in arid regions, but also in humid regions and frequently even in the lower horizons of soils with calcium as the principal exchangeable ion in the surface layers. While there is some justification on pedological grounds for considering the value at pH 8·4

as a better expression of the cation exchange capacity of the soil than that at pH7, it might be argued, on similar grounds, that pH 9·5, that of a system in which there is equilibrium between exchangeable magnesium, magnesium carbonate, and atmospheric carbon dioxide, or even the pH of the exchangeable sodium—sodium carbonate—carbon dioxide system, would be a more suitable general reference point.

Hissink (1924), in measuring the saturation or exchange capacity by titrating the soil with a solution of barium hydroxide, did imply a more alkaline reference point of about pH 10 or 11, as did Page (1926), although no definite point was actually expressed. Other investigators have determined the exchange capacities by titration methods at reactions greater than pH 8·4, but in most cases the end-points have been quite indefinite, since they have depended entirely upon the method or indicator employed. Indicators used have included *o*-cresol phthalein or phenol phthalein with an end-point about pH 9, or thymol phthalein with an end-point as high as pH 10. According to Bradfield and Allison's definition of a saturated soil, any soil with pH greater than 8·4 and a total content of exchangeable cations in excess of that at pH 8·4 would simply be considered as over 100 per cent. saturated.

From this brief summary of the present position it is evident that there is an urgent necessity for agreement to be reached, firstly on the definition of cation exchange capacity, which should be related as closely as possible to a fundamental property of the soil, and secondly on a method for its determination.

The object of the following investigation was therefore first to devise a simple and reliable method for the rapid determination of the cation exchange capacities over a range of pH values, and secondly, from the results so obtained to determine the most logical reference point for the characterization and comparison of the exchange capacities of soils. While such a point is of fundamental importance, a value of more practical significance so far as each individual soil is concerned is its exchange capacity at its own pH value. This value is a definite characteristic of the soil as it occurs in its natural state. Since, however, any treatment in the field will necessarily upset the balance in the extent and nature of the exchangeable cations present, and must result in some variation in the pH value, the pH of the soil may become somewhat transient, in the surface layers at least, under cultivation, and a knowledge of the likely variation in cation exchange capacity with change in pH is essential.

2. Materials Examined

As subjects for examination, 29 soils and 17 of the colloid fractions ($< 1\,\mu$) separated therefrom were chosen. They are representative of soils ranging from true podsols, in which leaching processes are active for most months of the year, and in which at no time is there a real moisture deficiency, to chestnut earths, in which leaching is extremely restricted and drought conditions persist for over six months of the year. Yellow and red-brown podsolized soils, red loams, red-brown earths, and black earths, as well as alluvial and immature profiles, are included; these are all soils in which intermediate leaching conditions prevail, and there is a progressive increase in droughty conditions in passing from the first to the last-named. The soils have

developed on a range of parent materials derived from granites, basalts, diabase, pumiceous ash, sedimentary shales, slates, and schists, and a ferruginous quartzite; the soil from the last-named is, however, better described as an ochreous iron ore than a true soil.

For the majority of the soils, the clay mineral types in the colloid fraction have been identified by X-ray examination, and this information, together with that given above and the figures for clay and organic carbon content and pH values for each of the soils considered, is given in Table 1. In the last column of the table reference is made to the literature, in which detailed descriptions of the soils concerned may be found. The soils are arranged in descending order of magnitude of their maximum cation exchange capacities, as subsequently determined.

Certain clay mineral samples which have already been described in detail by the author (Hosking 1942) have also been included.

3. Experimental

(*a*) *Method for Determination of Cation Exchange Capacities* *

Since ammonium acetate solutions with a wide range of pH values may be readily prepared by mixing ammonium hydroxide and acetic acid solutions in varying proportions, cation exchange capacities at any desired pH may be conveniently determined by extending the scope of the method embracing the leaching of the samples with a neutral normal solution of the salt (Hibbard 1939).

As a leaching agent, ammonium acetate has many advantages over other salts. In the first instance, the solution is strongly buffered, more so than that of the "other" alkali metal salts in the alkaline as well as the acid range, particularly in the vicinities of pH 5 and pH 9. Bower and Truog (1940) have shown that the magniture of the cation exchange capacity is definitely influenced by the valence of the cation employed in its determination; values obtained from the adsorption of divalent and polyvalent cations are significantly high, owing to the formation of basic "salts" with the exchange acids, and these values increase with the decreasing strength of the base which the respective cations form. The ammonium ion, since it is monovalent, will give normal values.

A further factor in favour of ammonium acetate is associated with the acetate ion. Since the latter is monovalent, no excessive adsorption of the ammonium ion will occur as with polybasic exchangeable anions such as the phosphate ion (Hosking 1942).

Finally, the estimation of the adsorbed ammonium ion, by direct distillation of the ammonia from the sample, may be carried out rapidly and in a single operation.

Preparation of the ammonium acetate solutions.—Ammonium acetate solutions, normal with respect to ammonium, may be prepared by mixing ammonium hydroxide and acetic acid solutions in the

*The method described is based upon procedures originally developed by the author (Hosking 1942) in collaboration with R. P. Stout (1939) in the Plant Nutrition Laboratory of the University of California.

Soil	Horizon	Locality	Zonal Soil Type	Parent Material	Clay Mineral Types§ of Clay Fraction	Clay <2μ	Organic Carbon	Reaction	Cation Exchange Capacity			References
									Soil		Colloid	
									At pH of Soil and Variation per Unit pH	Max. at pH 9	Max. at pH 9	
						%	%	pH	m.e.%‖	m.e.%	m.e.%	
1048*	B	Orion Downs, Qld. ..	Black earth ..	Basalt	M	62·0	1·46	7·4	86·6 ± 0·9	88·2	109·1	(1, 3)
1216*	B	Illparan, N.S.W. ..	Black earth ..	Basalt	M	50·6	1·71	6·7	64·8 ± 0·8	66·8	96·1	(1, 3)
5386	B	Rabaul, N.G. ..	Immature ..	Volcanic ash ..	K+?	11·0	0·77	7·0	..	..	89·3	(2, 3)
1007†	B	Gunnedah, N.S.W. ..	Red-brown earth ..	Basalt	M	55·3	0·93	8·1	48·5 ± 0·8	48·8	78·9	(3, 8)
Vina	A	California, U.S.A. ..	Alluvial	Basalt	K+?	..	..	6·6	..	..	66·4	(6)
Yolo	A	California, U.S.A. ..	Alluvial	Sandstone and shale	M	..	..	7·0	..	..	60·6	(6)
3719†	B_1	Tarlee, S.A. ..	Grey-brown soil‡ ..	Shales, slates, &c. ..	..	53·6	0·89	9·0	47·2 — 0·3	47·2	..	(7)
3715†	B_1	Riverton, S.A. ..	Red-brown earth ..	Shales, slates, &c. ..	..	60·5	0·84	9·1	45·0 — 0·6	45·1	..	(7)
1005†	A	Gunnedah, N.S.W. ..	Red-brown earth ..	Basalt	M + K	40·1	2·49	7·5	42·7 ± 0·65	43·4	..	(8)
3798†	AB	Booleroo, S.A. ..	Red-brown earth ..	Shales, slates, &c. ..	..	47·0	1·48	8·4	40·9 ± 0·9	41·3	..	(7)
6508*	A	Ulverstone, Tas. ..	Red loam ..	Basalt	K	38·1	5·00	5·3	30·5 ± 2·9	41·0	47·1	(9, 10)
4373*†	B	Denmark, W.A. ..	Red-brown podsolized	Diabase	M + K	37·6	0·40	5·1	33·9 ± 1·0	38·5	57·4	(3, 5)
1845	B	Mirrool, N.S.W. ..	Chestnut earth ..	Basalt	M + K	49·7	0·51	8·3	37·6 ± 0·7	37·8	60·1	(3)
3745†	B_1	Clare, S.A. ..	Red-brown earth ..	Shales, slates, &c. ..	..	61·6	0·79	7·7	30·5 ± 0·95	31·9	..	(7)
4370†	A_1	Denmark, W.A. ..	Red-brown podsolized	Diabase	M + K	12·1	4·16	6·5	27·7 ± 1·8	30·6	..	(5)
3946*†	A	Denmark, W.A. ..	Red-brown podsolized	Basic gneiss or diabase	K	16·4	2·66	6·5	25·5 ± 2·1	30·1	..	(5)
6509*	A	Cressy, Tas. ..	Red-brown podsolized	Clays and gravel ..	K	41·0	2·42	6·1	22·2 ± 1·0	27·5	32·4	(9, 11)
3759†	B_1	Saddleworth, S.A. ..	Red-brown earth ..	Shales, slates, &c. ..	..	41·7	0·30	7·3	22·3 ± 1·0	22·9	..	(7)
Aiken*	A	California, U.S.A. ..	Red loam ..	Basalt	K	..	..	..	..	..	25·7	(4, 6)
902†	B	Coolangatta, Qld. ..	Red loam ..	Basalt	K	63·7	1·81	5·2	16·8 ± 0·7	21·1	24·8	(3, 8)
1375	B	Wambanumba, N.S.W.	Red-brown podsolized	Granite	K	61·1	0·66	7·9	18·1 ± 1·0	19·1	25·8	(3)
3994*†	A_1	Denmark, W.A. ..	Yellow podsolized ..	Granitic gneiss ..	K	9·8	4·05	6·0	12·6 ± 1·4	18·4	..	(5)
3723†	A	Tarlee, S.A. ..	Red-brown earth ..	Shales, slates, &c. ..	..	21·5	0·83	7·9	14·5 ± 0·6	14·9	..	(7)
3496*	A	Waite Institute, S.A.	Red-brown earth ..	Shales, slates, &c. ..	K	17·4	1·43	6·4	11·7 ± 0·6	13·1	31·7	(7)
2187	B	King Island, Tas. ..	Podsol	Granite	K	34·0	1·06	6·6	..	..	20·0	(3, 12)
3948*†	B	Denmark, W.A. ..	Red-brown podsolized	Basic gneiss or diabase	K	35·4	0·54	7·2	..	6·6	9·5	(3, 5)
3948*†	B	Denmark, W.A. ..	Yellow podolized ..	Granitic gneiss ..	K	39·2	0·32	6·4	3·3 ± 0·4	4·6	9·8	(3, 5)
3401	B	Mundaring Weir, W.A.	Yellow podsolized ..	Granite	K	41·0	..	5·8	..	..	7·4	(3)
6560	A	Middleback S.A. ..	Iron ore	Ferruginous quartzite	G	9·3	..	..	..	7·0	7·4	..

* Cation exchange capacity also determined according to standard method with neutral ammonium acetate.
† Cation exchange capacity also determined by summation of cations at pH 8·4, see Fig. 1.
‡ In red-brown earth zone.
‖ M = clay mineral of the montmorillonite type, K = clay mineral of the kaolin type, G = predominantly goethite.
§ m.e.% = milli-equivalents per 100 g.

References.—(1) Hosking (1935); (2) Hosking (1938); (3) Hosking (1940); (4) Hosking (1942); (5) Hosking and Burvill (1938; (6) Kelley *et al.* (1939); (7) Piper (1938); (8) Prescott and Hosking (1936); (9) Shearer and Cole (1939-40); (10) Stephens (1937); (11) Stephens, Baldwin, and Hosking (1942); (12) Stephens and Hosking (1932.)

proportions shown in Table 2. Columns 2 and 3 show the quantities of ammonium hydroxide in gram-equivalents per 10 litres for preparing solutions of pH values from 4 to 11, while columns 4 to 8 indicate the volumes of appropriate reagents to take to prepare working quantities of 10 litres each of these solutions. The pH values of these stock solutions should be checked, and where there is appreciable departure from the value sought, aliquots should be titrated and the bulk samples adjusted accordingly.

TABLE 2.—AMOUNTS OF AMMONIUM HYDROXIDE AND ACETIC ACID SOLUTIONS REQUIRED FOR MAKING UP 10 LITRES OF AMMONIUM ACETATE SOLUTIONS OF DEFINITE pH VALUES

pH	Gram-Equivalents		Amounts of Solutions to be Taken				
	NH_4OH	Acetic Acid	Acetic Acid 17·5N	Acetic Acid 5N	Ammonium Acetate N pH 7	NH_4OH N	NH_4OH (0.880) 18·5N
			ml.	ml.	ml.	ml.	ml.
4·0	10·0	98·7	3,643	..	..	..	540
5·0	10·0	18·6	1,063	..	..	..	540
6·0	10·0	10·81	..	162	10,000*	..	..
7·0	10·0	10·00	..	..	10,000	..	..
8·0	10·0	9·80	..	..	9,800	200	..
8·4	10·0	9·34	..	..	9,340	660	..
9·0	10·0	8·33	..	..	8,333	..	90
10·0	10·0	0·654	..	..	854	..	505
11·0	10·0	0·118	..	..	128	..	534

* Final dilution 10,162 ml., the remaining solutions 10,000 ml.

Saturation of the soil with ammonium ion.—From 1 to 5 g. of the soil, depending upon the cation exchange capacity, or from 0·1 to 1·0 g. of the soil fraction or clay mineral, depending upon the amount of material available, is weighed out into a 1-oz. (30 ml.) narrow-neck pharmacy bottle, fitter with a screw cap to resist pressure; 22·5 ml. of the appropriate ammonium acetate solution is added, the cap screwed down, and the bottle shaken vigorously. The bottle is then placed in a water bath maintained at 60°–70°C. for two hours, and vigorously shaken every 15 minutes. After removal, the bottle and contents are allowed to stand overnight.

The suspensions are then filtered through a small Buchner funnel fitted with a 4·25 cm. Whatman No. 44 filter paper, first moistened with the same solution as used for leaching, using slight suction. For small samples, generally 0·5 g. or less, or for coarse or sandy samples, for which subsequent filtration may be too rapid, Gooch crucibles fitted with 1·8-cm. papers should be used. Where the initial filtrate is turbid it must be returned to the filter before proceeding.

The bottle and cap are rinsed with 10 ml. and then a jet of the leaching solution from a wash bottle; the rinsing is repeated with small lots of the solution till the sample is transferred quantitatively to the filter. The leaching is continued with 5 to 10 ml. portions of the solution till the total leachate amounts to 100 ml. in all. The sample should be allowed to drain completely between each addition.

Exchangeable cations if desired may be determined in the filtrate.

The receiving flask is replaced and the excess of free ammonium salt remaining in the sample is leached out with 5 to 10 ml. portions of 60 per cent. alcohol, washing down the material adhering to the sides of the filter each time with a fine jet of alcohol from a wash bottle. A total of 50 ml. is usually sufficient. Finally the sample is rinsed with 2 lots of 5 ml. of absolute alcohol and allowed to just dry out. Prolonged exposure of the sample to the air current of the filter after the sample is dry should be avoided owing to possible losses of ammonia.

Determination of ammonia.—Direct quantitative transfer of the small sample to the distilling flask is difficult, and it is advisable to transfer it first to a small beaker from which it may be washed into the flask. When the sample is dry, carefully invert the Buchner funnel or Gooch crucible over a small beaker (100 ml. squat) and allow the sample and filter paper to drop into the beaker. Wash the funnel with distilled water to remove the last traces of the sample, rubbing it up if necessary with a rubber-tipped rod. Then transfer the suspension together with the filter paper into the distilling flask. Sufficient 0·1N hydrochloric acid should be placed in the distilling flask before adding the ammonium-saturated sample to prevent any loss of ammonia while standing in the flask.

The suspension is diluted to 250 to 300 ml. with distilled water, and to this is added either (*a*) 0·5 g. MgO for soils or samples containing organic matter, or (*b*) 25 to 30 ml. of N NaOH for clay minerals or soil fractions free from organic matter. About 200 ml. is distilled off in a Kjeldahl apparatus, and the ammonia collected in standard hydrochloric acid: (i) 0·05N for samples over 1g. in weight, or (ii) 0·02N for samples of 1 g. or less. The excess acid is titrated with standard sodium hydroxide, 0·025N in the case of (i), 0·01N in the case of (ii), using methyl red, sofnol red, or preferably a mixture of methyl red and methylene blue as indicator. In the titration with dilute sodium hydroxide, the end-point is not so well defined as with normal solutions, and a blank titration should be run with each set of determinations, and the end-point for each titration carefully compared with this as standard.

Cation exchange capacity.—The cation exchange capacity of the sample at any particular pH value, in milli-equivalents per 100 g., is then equal to:

$$\frac{100\ N\ (B-T)}{W} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where N = the normality of the standard NaOH, B = ml. of NaOH for the blank determination, T = ml. of NaOH in the actual titration, and W = weight of the sample in grams.

The cation exchange capacity of the soil at its own pH value may be read off from the curve connecting pH and exchange capacity.

(b) Consideration of the Method and its Possible Range of Application

The results for a range of typical soils, soil colloids, and clay minerals are given in Table 3 whereas those for all the samples examined are illustrated graphically in Fig. 1. For all the samples

the exchange capacities are seen to increase steadily from a minimum value at pH 5 to a maximum value at pH 9 beyond which they decline, some rather rapidly, with further increase in the reaction to pH 10 or 11. Many of the samples were peptized appreciably at pH 11 with the result that filtration was impossible and the determination of the capacity out of the question.

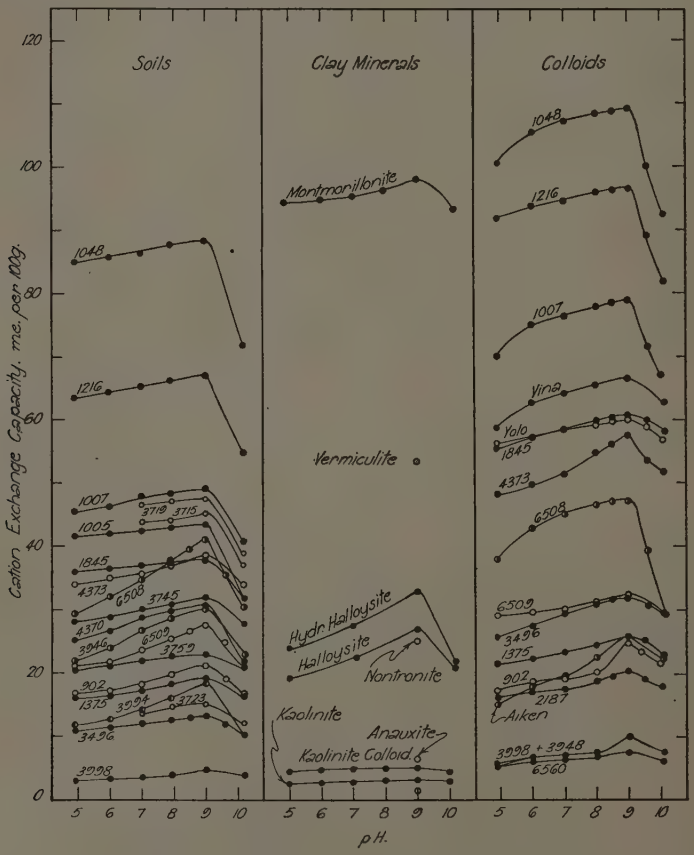

FIG. 1.—Showing the variation of the cation exchange capacity with pH, for the soils, soil colloids and clay minerals investigated.

From Table 3 it is seen that the values at pH 4 are frequently higher than those at pH 5 and it is probable that at this pH the acetate is adsorbed and so the capacity rises. Actually the solution at pH 4 is about 10N in acetic acid, and there is an appreciable attack on the mineral material of the samples; a loss of 2 per cent. (0·7 per cent. SiO_2, 1·0 per cent. Al_2O_3, and 0·3 per cent. Fe_2O_3) was

TABLE 3.—VARIATION IN CATION EXCHANGE CAPACITY OF SOILS. SOIL COLLOIDS AND CLAY MINERALS WITH pH

Sample	Soil Number or Mineral Sample	Cation Exchange Capacity at pH—								
		4	4·9	6	7	7·9	8·5	9	9·6	10·2	10·9
		m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	
Soils	1048	..	85·0	85·8	86·2	87·6	..	88·2	..	71·6	..
	1216	..	63·4	64·4	65·0	66·4	..	66·8	..	54·8	..
	1007	..	45·4	46·1	47·8	48·4	..	48·4	..	40·5	..
	6508	30·0	29·3	32·2	34·6	38·1	39·4	41·0	37·0	30·2	25·6
	3745	..	28·1	28·7	30·3	30·6	..	31·9	..	27·6	..
	6509	21·5	21·3	21·9	23·5	25·5	26·6	27·5	25·0	20·5	17·9
	902	..	16·7	17·4	18·2	19·7	..	21·1	..	16·8	..
	3496	10·1	10·8	11·4	12·1	12·5	13·0	13·1	12·1	10·3	7·5
	399	2·8	3·0	3·2	3·5	3·7	..	4·6	..	3·8	3·0
Soil colloids	1048	108·1	100·6	105·8	107·0	108·3	108·9	109·1	100·4	92·9	76·3
	1216	95·5	91·8	93·8	94·6	96·0	96·2	96·5	89·2	81·9	70·2
	1007	77·8	69·9	75·5	76·3	77·8	78·4	78·9	71·5	67·1	51·2
	Yolo	54·4	55·4	57·1	58·5	59·8	60·2	60·6	60·0	58·3	52·8
	6508	38·8	37·8	42·8	45·0	46·5	..	47·1	39·3	29·2	21·0
	3496	23·3	25·9	28·0	29·3	30·8	31·6	31·7	31·6	29·7	25·0
	Aiken	14·6	15·0	17·8	19·5	21·1	..	25·7	..	22·3	..
	902	17·5	17·3	18·8	19·0	20·0	..	24·8	23·2	21·9	18·1
	3998	5·0	5·1	6·9	7·0	7·2	..	9·8	..	7·6	5·6
Clay and related minerals	Permutite	264	284	347	347	347	..	349	..	348	345
	Montmorillonite	104·1	94·3	94·8	95·3	95·8	..	98·1	..	93·7	68·3
	Hydrated halloysite* ..	..	23·8	..	27·6	..	..	32·7	..	21·7	..
	Halloysite	..	19·2	..	22·3	..	..	26·7	..	20·8	..
	Kaolinite <1μ	4·4	4·4	4·6	4·8	4·8	..	4·8	..	4·4	2·8
	Kaolinite <2mm. ..	2·8	2·8	2·8	3·1	3·1	..	3·1	..	3·0	2·8

* "Endellite" of Alexander *et al.* (1943).

found for the Aiken colloid sample. From pH 5 to 9 the attack on the clay mineral was negligible but it increased somewhat at pH 10, and at pH 11 it was again appreciable and of the same order as at pH 4.

Methods for the determination of cation exchange capacity have been standardized for reaction values at pH 7 and 8·4 and hence the reliability of the present method may be assessed, at least for these pH values, by comparing the results obtained in the present work with those obtained according to these standard procedures.

Ten grams of each of the soils marked with an asterisk in Table 1 were treated according to the standard method of leaching with neutral normal ammonium acetate (Hibbard 1939); in addition a series of samples of the Aiken colloid from 0·1 g. to 5 g. were treated in a similar manner. The cation exchange capacities thus determined were compared with the results at pH 7 determined in the present work. Few of the results differed by more than 0·5 m.e. per 100 g., either for the soils or the various fractions of the colloids, the order of the differences being the same as that between the duplicates for either procedure.

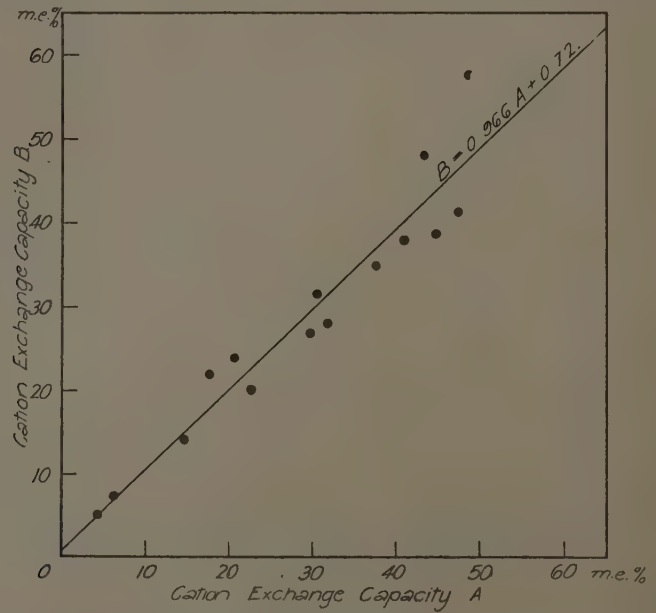

FIG. 2.—Comparison between cation exchange capacities of soils at pH 8·4 obtained by the present method (A) and the method embracing the summation of exchangeable cations (B).

The exchange capacities determined at, or calculated for, pH 8·4 in these investigations were compared with the results obtained by summation of the exchangeable metallic and hydrogen cations, according to the procedure recommended by Piper (1944), for the soils

marked with a dagger in Table 1 (see Fig. 2), and a very high degree of correlation (R = 0·96) is indicated. The regression equations—

$$B = 0{\cdot}966a + 0{\cdot}72 \text{ or } A = 1{\cdot}0356b - 0{\cdot}74 \ldots . \quad (2)$$

(where A,a represent the exchange capacities according to the present method and B,b the values by summation of the contributing cations), have been calculated and the mean of the regression lines is shown in Fig 2. Atlhough some divergence between the values is natural it is not possible to state definitely which of the values is to be preferred: a point in favour of the present method, however, is that only a single determination is concerned therein, whereas in the summation method at least five separate estimations are necessary and hence there is more likelihood of error in the latter procedure.

Outside the range pH 7 to 8·4, determinations of cation exchange capacity have been limited and there are no standard methods with which further comparison may be made. However, over the range from pH 5 to 9 the steady increase in the capacity would appear normal from ordinary physical chemical considerations, but the sudden decline with the increase in pH above 9, particularly for the colloid samples, was unexpected.

Part of this reduction in the capacities of the soils at pH 10 and 11 can be accounted for by the solubility of the organic matter at these alkaline reactions. Over the range from pH 4 to 9 the ammonium acetate leachates were quite clear but at the higher reactions they were very dark in colour; for some soils even at pH 10 the colours of the residual soils after leaching indicated an almost complete removal of all but essentially woody organic matter.

To obtain a measure of the reduction in the exchange capacity due to this loss of organic matter, seven soils were treated with hydrogen peroxide in order to remove the organic matter entirely. The exchange capacities of the residues were then determined over the whole reaction range from pH 5 to 11, and in Fig. 3 the results are compared graphically with those for the normal soils. From pH 5 to 9 it will be seen that the two curves for each of the soils before and after treatment, run more or less parallel, but beyond pH 9 they converge, and for three of the samples coalesce at pH 10 and for the remainder at pH 11. The distance between the curves is undoubtedly a measure of the contribution from the organic matter to the total exchange capacity. That this distance remains virtually constant up to pH 9 and then gradually decreases beyond this reaction indicates clearly that the alkalinity of the leaching solution in the latter region causes an appreciable solution of the organic matter.

However, these results for the residues free from organic matter only serve to emphasize that the losses of organic matter account for only a very small proportion of the decreases in the exchange capacities beyond pH 9. Two possible reasons for these decreases in the case of the mineral materials were considered, the one being associated with the decomposition of the clay minerals, and the second a possible irreversible precipitation of exchangeable calcium (the colloid samples were all originally calcium-saturated) as calcium hydroxide, within or upon the surfaces of the minerals at these alkaline reactions.

To test the first point, a sample of each of the clay minerals montmorillonite and kaolinite and seven soil colloids, with a range in exchange capacities from 4 to 100 m.e. per 100 g., were leached with ammonium acetate solutions, in one set at pH 10 and in the other

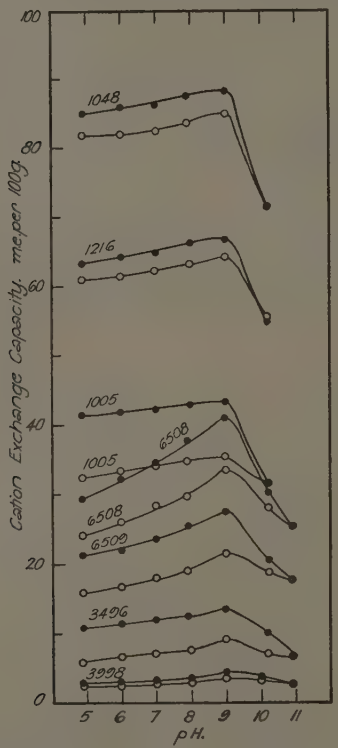

FIG. 3.—Variation of cation exchange capacity with pH for soils (dots) and soils freed from organic matter (open circles).

at pH 11. The exchange capacities of these treated samples were then determined in the ordinary way at pH 9. These results when compared with those for the normal samples (see columns 2 to 4, Table 4) show that any decomposition which may have taken place has little effect on their exchange capacities.

That the presence of exchangeable calcium is equally without effect is shown by the figures in colums 5 to 8 in Table 4. Seven of the above clay minerals and colloids were leached with ammonium acetate at pH 6 to remove the calcium present before determining the exchange capacities at pH 10 and 11. In each case the resultant capacities are practically the same as the capacities for the norml samples.

TABLE 4.—CATION EXCHANGE CAPACITIES OF SOILS AT pH 9 AFTER LEACHING WITH AMMONIUM ACETATE SOLUTIONS OF pH 10 AND 11, AND AT pH 10 AND 11 AFTER LEACHING WITH AMMONIUM ACETATE OF pH 6

Sample	Cation Exchange Capacity at—						
	pH 9			pH 10		pH 11	
	Normal	Sample Leached at pH 10	Sample Leached at pH 11	Normal	Sample Leached at pH 6	Normal	Sample Leached at pH6
Montmorillonite ..	98·1	98·4	98·7	93·7	92·1	89·8	89·8
Vina colloid ..	66·4	64·6	65·3	62·9	61·4	57·6	60·2
1843 colloid ..	60·1	62·6	63·6	57·3	56·7	46·0	45·6
6508 colloid ..	47·1	46·6	44·5	29·2	29·3	..	..
6509 colloid ..	32·4	33·6	33·8	29·5	29·4	..	..
3496 colloid ..	31·7	33·6	33·9	29·7	30·0	..	..
1375 colloid ..	25·8	26·2	26·4	23·9	22·1	..	..
3998 colloid ..	9·8	10·0	10·1	..	..	..	..
Kaolinite colloid ..	4·8	5·0	5·1	..	..	..	..

Although further work is required * to explain fully these decreases in the cation exchange capacity above pH 9 there is no doubt that the decreases are real. In any case the solubility of the organic matter in solutions more alkaline than pH 9 precludes an accurate determination of the exchange capacity for soils at these reactions, although this does not apply for soil colloids and clay minerals. For a soil with a natural pH value higher than 9, of course, the soil organic matter present would necessarily be in equilibrium with conditions pertaining to the particular pH and hence no appreciable loss of organic matter would occur when leaching with a solution of that reaction.

Although the figures over the acid range are of the type and order one might expect in the light of those obtained at pH 7 and 8·4, further work might be undertaken, in the absence of comparative data over this range, to substantiate their absolute accuracy. However, the data obtained for the range of pH values represent a first approach to the investigation of the problem outlined and are thus of definite intrinsic value.

A survey of the results indicates that pH 9 assumes a definite significance as the pH value at which a maximum cation exchange capacity occurs.

So far as the method itself is concerned it may be used for determining, in a single operation, exchange capacity of soils over the useful range from pH 5 to 9 and beyond this for mineral samples. It has the advantage over other methods that only small samples, of the order of as low as 0·1 to 0·5 g., may be used where material is restricted, and it is extremely rapid; from 90 to 100 determinations can be conveniently handled in a week.

* Investigations along these lines were abandoned at the time owing to the transfer of the author from the Division of Soils.

4. Discussion of Results

Reference to Table 3 or Fig. 2 indicates that the sample of montmorillonite examined has a maximum capacity of 98 m.e.*, a value of the order (from 80 to over 100 m.e.) usually found for samples of the mineral. From a minimum of 95 m.e. at pH 5, the capacity increases only slowly by about 1·0 m.e. per unit pH to the maximum value at pH 9, and then falls away sharply to 94 m.e. at pH 10 and even more rapidly to only 68 m.e. at pH 11.

Both the kaolinite and the colloid separated therefrom have extremely low maximum exchange capacities of 3 and 5 m.e. respectively, values somewhat lower than the mean value of about 8 m.e. (at pH 7) usually taken as a representative value for kaolonite. Although by no means pronounced, there is apparently a real increase from the minimum capacities of 2·8 and 4·4 m.e. respectively at pH 4, to maxima of 3·1 and 4·8 m.e. at pH 9 for the kaolinite and its colloid; tendencies towards a decrease in both cases are noticeable when the reaction is increased to pH 10 and 11.

The capacities for the hydrated halloysite are from 8 to 10 times as high as those for the kaolinite, and the increases over the range from pH 5 to 9 are more pronounced than for the montmorillonite. The exchange capacity rises from a minimum of 24 m.e. at pH 5 to a maximum of 33 m.e. at pH 9 with an increase in the capacity per unit pH of about 2 m.e. The halloysite, while showing the same general increase in the capacities up to pH 9 and the same pronounced drop in the value at pH 10, has somewhat lower capacities over the whole range.

The sample of permutite or artificial zeolite has a capacity about three times that of montmorillonite. Although the values are somewhat lower at reactions more acid than pH 6, the capacity of about 350 m.e. remains fairly constant over the range from pH 6 to 11, with a probable maximum at pH 9, as for the clay minerals. The general shape of the curve, however, is significantly different from that of the clay minerals in that there is no appreciable falling off in the capacity beyond pH 9.

Although quantities available did not allow of a more extended examination, maximum cation exchange capacities have been determined for a few other minerals. For talc and pyrophyllite the values of 1·4 and 1·3 m.e. respectively were negligible, the value 6·5 m.e. for anauxite is of the same order as for kaolinite; a coarsely crystalline sample of nontronite has a capacity of only 23·4 m.e., and finally for a sample of vermiculite the maximum exchange capacity is 53.6 m.e.

The results for the individual soils and soil colloids indicate that in general those containing a clay mineral of the montmorillonite type have the highest capacities, although the samples characterized by a mineral of the kaolin type have capacities which are much higher than might be expected. Whereas it has been shown that the mineral kaolinite has an extremely low capacity, soil colloids in which only a mineral of the kaolin type has been identified have capacities ranging

* In this section for m.e. read the full expression "milli-equivalents per 100 g."

from 7 m.e. for sample 3401 to as high as 66 m.e. for the Vina colloid from California (compare the figure given by Kelley *et al.* 1939) and even 89 m.e. for the colloid from soil 5386 from New Guinea. While it is possible for as much as 10 per cent. montmorillonite to be present in a sample without its detection by X-ray methods of examination, the presence of a clay mineral of this type would appear to be unlikely in many of the samples, when consideration is given to other factors such as the chemical analysis. In any case, such a small percentage of the mineral would only contribute some 10 m.e. or less to the capacity.

While exchange capacities of the order of from 25 to 30 m.e. may, in the case of the colloids from the Aiken soil and soils 1375, 902, and 6509, be related to the probable presence of the mineral hydrated halloysite or halloysite, the capacities of the Vina colloid and soil 5386 cannot be explained in this way. For the two last-named colloids the X-ray patterns were of a general low intensity, and the kaolinite patterns obtained were somewhat imperfect, thus indicating a poor degree of crystallinity for the material as a whole. Although a small percentage of montmorillonite may be present, amorphous materials with exchange capacities of a very high order, possibly as high as that of the permutite, may be the principal contributors to these reactions. It is more than likely also that amorphous materials with high cation exchange capacities play a much more important role in other soils as well than has been attributed to them in recent years.

All the soil colloids show the pronounced maximum in the cation exchange capacity at pH 9, with a rate of increase, over the pH range from a minimum at pH 5 to this value at pH 9, quite independent of the type of clay mineral or the magnitude of the capacity. For the colloids, the rate of increase in the capacity per unit pH varies from 0·8 m.e. for 6560 to 2·7 m.e. for the Aiken colloid. Five colloids show increases of about 1·0 m.e.; these include 3998 with a maximum exchange capacity of 7·4 m.e. and characterized by a kaolin mineral, and 1845 with a maximum capacity of 60 m.e. containing montmorillonite essentially, although a little kaolinite is also present. Seven colloids, including Nos. 1048 and 1216 containing only montmorillonite and with maximum exchange capacities of 109 and 96 m.e. respectively, and 902 containing only kaolinite (21 m.e.), show increases ranging from 1·0 to 2·0 m.e. per unit pH. The remaining three colloids with exchange capacities from 26 to 57 m.e. show increases of up to 2·7 m.e. per unit pH.

In some of the colloids there is a sharp initial rise between pH 5 and 7 with a less pronounced increase between pH 7 and 9; in others the reverse is the case, but generally speaking the increase in the exchange capacity is more or less uniform over this reaction range. Although all the colloids show a sharp decrease at pH 10 and 11, the decrease is particularly marked for the montmorillonite colloids.

The exchange capacities of the soils are definitely lower although generally in much the same order as those of their colloids; where marked departures from the order occur they may be related to pronounced differences in clay content. The presence of organic matter

in reasonably high concentrations does not materially offset the smaller capacities of the soils owing to the admixture with the coarser inert material.

The montmorillonitic black earths 1048 and 1216 show the highest maximum cation exchange capacities of 87 and 64 m.e. respectively, while the kaolinitic yellow and red-brown podsolic soils have the lowest values of from 4 to 13 m.e. The red-brown earths show a wide range of capacities from the highest value of 49 m.e. for soil 1007 from Gunnedah in New South Wales with a high proportion of montmorillonite (montmorillonite to kaolinite = about 8:1 in the colloid) to the lowest, 13 m.e., for soil 3496, a soil from the Waite Institute, South Australia, in which kaolinite is apparently the only clay mineral type. The remaining soils, including the chestnut earth 1845, the red loams, and red-brown podsolized soils, show maximum exchange capacities varying from 20 to 40 m.e.; the lower capacities are definitely associated with kaolinite colloids, while the higher ones are related to a content of montmorillonite in excess of the kaolin minerals.

The increase in the exchange capacities per unit pH for the soils is generally of the same magnitude as that for the colloids, with the rate of increase again independent of the type of clay mineral or the magnitude of the capacity. Organic matter apparently affects the rate of increase but little, as may be observed from the curves in Fig. 3. Soil 6508 shows the highest overall increase of about 3 m.e. per unit pH in passing from pH 5 to 9. In this soil the organic matter amounts to 9 per cent., and a definite increase in the capacity per unit pH due thereto is of the order of about 0·5 m.e.

The smallest increase of 0·5 m.e. per unit pH is shown by soils 1005, with a maximum exchange capacity of 43 m.e., and 3998, with a maximum capacity of only 5 m.e. One soil, 3946, shows an increase of 2 m.e., three soils an increase of 1·5 m.e., while the remainder show increases ranging from only 0·7 to 1·5 m.e. per unit pH.

It is clear from Fig. 2 that the curves for the soils are of much the same general shape, although the results for soils such as 3946, 6508, and 6509 stand out somewhat from the remainder.

From these curves the cation exchange capacities of the soils at their own pH values have been determined and are given along with the maximum exchange capacities in Table 1. In addition, the variation in the exchange capacity with either unit increase or decrease in pH from that of the soil is shown; where the pH of the soil is in the neighbourhood of pH 5, only the increase is shown, and similarly where the pH is in the vicinity of 9, only the decrease is given, owing to the uncertainty of the values obtained at pH 4 and 10.

Since these capacities are of fundamental significance it is suggested that the term "cation exchange capacity of the soil" should be reserved for the capacity at the pH value of the soil in its natural state, i.e. at its own pH, and the term "maximum cation exchange capacity" should be used for that at pH 9. This latter value can be used where a comparison of the exchange capacities of different soils, colloids, and clay minerals is required. Ready comparison with

capacities for soils already expressed at pH 7 and 8·4, or any other pH for that matter, is possible by reference to the curves covering the comprehensive range.

It is suggested that, for complete characterization of the exchange reactions of soils, the maximum exchange capacity, the cation exchange capacity of the soil (at its own pH), and the magnitude of the variation per pH unit with increase or decrease in pH from that of the soil should be given. This has been done in the present work, and is intended to give a lead for future work in this field.

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The Cation Exchange Capacity of Soils and Soil Colloids

2. The Contribution from the Sand, Silt, and Clay Fractions and Organic Matter

*By J. S. Hosking, M.Sc., Ph.D.**

Summary

Cation exchange reactions are shown to be by no means confined to the clay or colloid fractions of soils. Although there is admittedly a decline in the cation exchange capacity in passing from the colloid to the silt fraction, the capacity of the latter is appreciable and persists into all but the most highly siliceous sand fractions. The exchange capacities of the silt and sand fractions are shown to be related to the presence therein of high concentrations of the clay minerals, which are distributed quite generally throughout all the fractions of the soil, as separated by standard procedures of mechanical analysis.

On the basis of the cation exchange capacities of the various fractions and the mechanical analysis of the soil, the capacity of the mineral portion of a number of soils has been calculated, and excellent agreement is obtained in each case with the value determined directly on the soil freed from organic matter.

Finally, by taking into account the total contributions from all the inorganic fractions, a value has been determined for the exchange capacity of the organic matter of the soil; the new average value of 280 m.e. per 100 g. of organic carbon is shown to be of the order of only one-half to one-quarter of the capacities previously suggested.

1. Introduction

It has long been considered that in addition to organic matter the only other materials contributing to the cation exchange capacity of soil are the clay minerals assumed to be concentrated in the colloid or clay fraction; the coarser silt and sand fractions have been considered as virtually inert.

Based upon this assumption and in the absence or direct determinations, Williams (1932) in England, and Prescott and Hosking (1936), Hosking and Burvill (1938), and Piper (1938) in Australia have calculated possible values for the exchange capacities of the clay fractions and the organic carbon by dividing, either by algebraic or by graphical methods, the total cation exchange capacity of the soil betwen these two components. Recent direct determinations on the clay fractions separated from certain of the soils (Hosking 1948), considered by the Australian authors, however, show that the estimated values for the clays are too high. Similarly, from the differences between the exchange capacities of the soils themselves and those of the mineral portions of the soils alone, i.e. the soil freed from organic matter by treatment with hydrogen peroxide, it is evident that the values given for organic matter have also been overestimated.

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Furthermore, when the cation exchange capacities of the mineral portions of a series of soils were compared with the values calculated from the capacities of their colloid or clay fractions, on the basis of the percentage of the clay present in the soil, the calculated values fell far short of the actual figures. The calculated value for one soil with an exchange capacity of 34 m.e. per 100 g. (see Table 3, soil 6508) was low by no less than 15 m.e.

It was quite apparent that the coarser silt and sand fractions must contribute materially to the total exchange capacity, and the following investigation shows definitely that this is so; in comparison, the cation exchange capacity of the organic matter must be reduced considerably by at least the amount due to these sources.

2. Soils Examined

The soils for examination were chosen from those already described in the previous paper (Hosking 1948, p. 21) for which the data are given for the colloid fraction. They represent a series of black earths, chestnut earths, red-brown earths, red loams, and red and brown podsolized soils in which the clay minerals of the montmorillonite and kaolinite types separately or together have been identified in their colloid fractions. The parent materials of the soils are predominently basaltic or granitic, but soils formed over diabase, shales and slates, clays and gravels, pumiceous ash, and ferruginous quartzite are also represented.

3. Preparation of the Fractions

For the separation of the various inorganic fractions sufficient amounts of the various soils were taken to ensure adequate quantities of each of the fractions for subsequent examination. Following treatment of the suspensions of the soils with hydrogen peroxide, adjusted to pH 5 where necessary, to ensure its efficient action in the destruction of the organic matter (Hosking 1932), the soils were dispersed and the procedure as described for ordinary mechanical analysis (Piper and Poole 1929) continued to the stage of the separation of the coarse sand ($> 0{\cdot}18$ mm.).

The dispersed soil suspensions were then transferred to tall beakers and the colloid fraction ($<1\mu$) was separated in the usual way by repeated decantation, following sedimentation of the soil through 8·6 cm. in 24 hr., at a mean temperature of 20°C. To separate the further fractions, coarse clay (2μ to 1μ), fine silt (10μ to 2μ), coarse silt (20μ to 10μ), very fine sand (40μ to 20μ), and fine sand (0·18 mm. to 0·04 mm.), the process of decantation was continued with sedimentations through 10 cm. at 20°C., for periods of time appropriate to the separation of such fractions. Sedimentations were continued until the amounts of material remaining in suspension were reduced to negligible amounts. The Aiken colloid was fractionated according to the method previously described by the author (Hosking 1944).

The separated fractions were flocculated where necessary with calcium chloride and, following the siphoning off of the supernatant liquid, treated with dilute acid to remove calcium carbonate and again (including the coarse sand fraction) with hydrogen peroxide to ensure

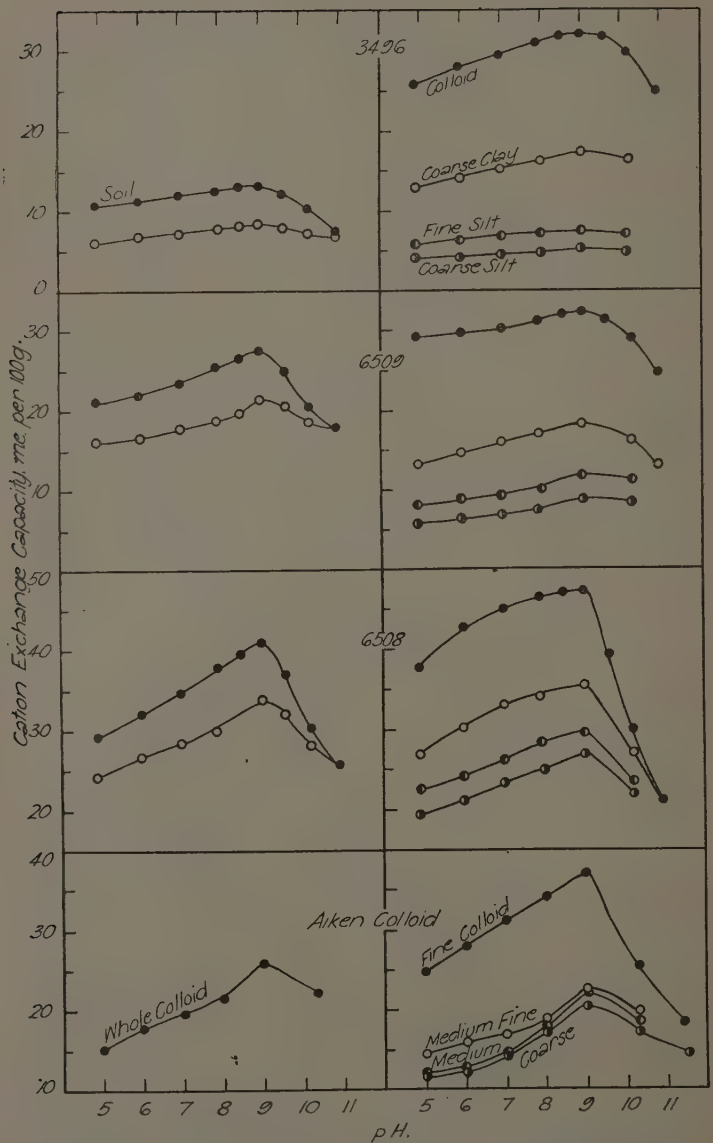

FIG. 1.—Showing the variation of the cation exchange capacity with pH of four fractions of the soils 3496, 6508 and 6509 and the Aiken colloid. For comparison the results for the soils before (dots) and after removal of organic matter (open circles) and for the unfractionated Aiken colloid are also shown.

complete freedom from organic matter. The samples were then filtered, leached with calcium chloride to ensure calcium saturation, washed with alcohol to remove excess salts, and air-dried. After weighing, and crushing to pass a 1 mm. sieve, the samples were finally brought to equilibrium in an atmosphere of 50 per cent. relative humidity.

The particle size distribution determined from the weights of the various fractions agreed very closely with the results for ordinary mechanical analysis.

4. Method for Determination of Exchange Capacity

The method for determination of the capacities was that described in the previous paper (Hosking 1948). Since in most cases the samples were limited, the determinations were confined almost entirely to that of the maximum exchange capacity at pH 9. For three of the soils, 3496, 6508, and 6509, the samples were adequate to determine the exchange capacities over the reaction range from pH 5 to 11. These latter results for the colloid, coarse clay, fine silt, and coarse silt fractions, together with those for the colloid subfractions of the Aiken colloid, are shown in Fig. 1.

The maximum cation exchange capacities for all the soil fractions, together with those for the soils before and after removal of the organic matter, are shown in Table 1, while in Fig. 2 the variations in the capacities with particle size are illustrated graphically.

5. Cation Exchange Capacities of the Sand and Silt Fractions

The results indicate very definitely that cation exchange reactions are by no means restricted to the colloid or clay fractions.* In most soils there is admittedly a sharp decline in the exchange capacities in passing from the colloid through the coarse clay to the silt fractions, but the capacities of the latter are quite appreciable and persist at much the same level into all but the most highly siliceous fine and coarse sand fractions. For the two soils 1048 and 1216 the exchange capacities of the sands and silts are appreciably higher than the capacities of most of the other colloids examined. In three of the soils, Nos. 1048, 6509, and 3496, each characterized by a different clay mineral in the colloid fraction—a montmorillonite type in the first, possibly halloysite rather than kaolinite in the second, and kaolinite in the third—the cation exchange capacities for the coarse sands are of the same order as those for their respective coarse clays.

An examination of Fig. 1 shows that the same general relationships between the cation exchange capacity and pH exhibited by the colloids holds for the coarser fractions. In all the samples examined the maximum capacity occurs at pH 9. With increase in particle size, however, there is a gradual decline both in the rate of increase in the capacity up to pH 9 and in the rate of decrease beyond this reaction.

* As early as 1920, Ogg and Hendrick (1920) showed that the fine silt and silt fractions of certain Scottish soils had an appreciable capacity for cation adsorption though the presence of organic matter in the fractions did not enable the determination of the exchange capacities of the inorganic materials.

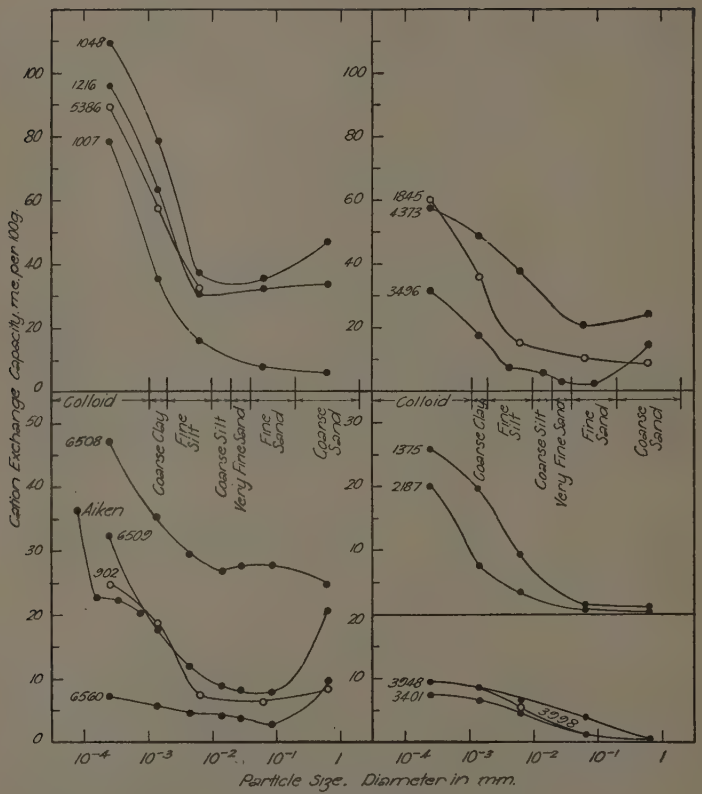

FIG. 2.—Variation of cation exchange capacity with particle size of the soils investigated.

This is not particularly marked for the fractions of soil 6509, where the capacities of all fractions are relatively high, but for soil 3496 where the capacities of the silt fractions are quite low the curves become appreciably flattened. In any one soil it would appear that the rate of increase in the capacity is related to the magnitude of the capacity or alternatively to the percentage of the exchange material present in the fraction. Although there is a pronounced difference between the exchange capacities of the fine and the three coarser fractions of the Aiken colloid, the shapes of the curves indicate somewhat similar rates of increase over the range from pH 5 to 9 for them all.

Since these exchange reactions are undoubtedly related to the presence of clay minerals in all the various fractions, it is clear that the clay minerals are not confined to the clay fractions, and this has been substantiated by a survey of the literature and an examination of the coarse sands during the present work.

TABLE 1.—MAXIMUM CATION EXCHANGE CAPACITY AT pH 9 OF THE VARIOUS FRACTIONS OF THE SOILS EXAMINED

Soil	Zonal Soil Type	Parent Material	Clay Minerals Type of the Colloid Fraction	Soil Fraction							Soil	
				Clay		Silt		Sand			Normal	Mineral Fraction
				Colloid	Coarse	Fine	Coarse	Very Fine	Fine	Coarse		
				<1·07μ	<1·07μ to 2·0μ	2·0μ to 10μ	10μ to 20μ	20μ to 40·5μ	40·5μ to 0·18mm.	0·18 mm. to 2·0 mm.		
				m.e. %	m.e. %	m.e. %	m.e. %	m.e. %	m.e. %	m.e. %	m.e. %	m.e. %
1048	Black earth	Basalt	Montmorillonite	109·1	79·0	37·9		35·3		47·0	88·2	85·5
1216	Black earth	Basalt		96·5	63·1	30·7		32·0		33·8	66·8	64·3
1007	Red-brown earth	Basalt	Montmorillonite+ kaolin	78·9	35·3	16·2		8·0		6·0	48·8	..
1845	Chestnut earth	Basalt		60·1	35·6	15·7		10·0		8·8	37·8	..
1373	Red-brown podsolized	Diabase		57·4	48·7	38·0		20·5		24·1	38·5	..
5386	Immature	Pumiceous ash	Kaolin	89·3	57·7	32·7		..	..	..	..	..
6508	Red Loam	Basalt		47·1	35·2	29·5	26·7	27·7	27·6	24·7	41·0	33·9
6509	Red-brown podsolized	Clays and gravel		32·4	17·8	11·7	8·8	8·3	7·8	20·6	27·5	21·6
3496	Red-brown earth	Shales, slates, and schists		31·7	17·3	7·5	5·2	3·0	2·2	14·8	13·1	9·4
1375	Red-brown podsolized	Granitic		25·8	19·9	9·1		1·4		1·1	19·1	..
Aiken	Red loam	Basalt		25·7	..	..		..		..	..	..
902	Red loam	Basalt		24·8	19·0	7·5		6·5		8·4	21·1	..
2187	Podsol	Granite		20·0	7·3	3·3		0·6		0·3	..	..
3998	Yellow podsolized	Granitic gneiss		9·8	8·5	5·3		0·7		0·1	4·6	3·7
3948	Red-brown podsolized	Basic gneiss or diabase		9·5	8·5	6·4		3·7		0·4	6·6	..
3401	Yellow podsolized	Granite		7·4	6·6	4·5		0·8		0·2	..	..
Kaolinite	Mineral sample			4·8	..	..	..	..	..	..	..	3·1
6506	Iron ore	Ferruginous quartzite	Goethite, haematite	7·4	5·9	4·4	4·2	3·8	2·7	9·6	7·0	..

A survey of the literature dealing with the mineral compositions of the silt and sand fractions of soils, particularly that relating to the fine sand fractions of Australian soils (Carroll 1931-2, 1933-4) shows that clay minerals have been definitely recognized in these fractions, sometimes dominating all other mineral types. In not every case, however, have the clay minerals been specifically identified; Carroll for instance has simply characterized the materials observed as either "kaolinized material" or "nontronite"; the former actually embraced montmorillonite and kaolin minerals as well as amorphous materials, whereas "nontronite" appears to include only minerals of the montmorillonite type. Marshall (1935) has examined in some detail the silts and sands from a bentonite and three soils. In the first-named the presence of from 30 to 50 per cent. of montmorillonite was recorded in the silt and about 3 per cent. in the sand fraction. In the silts and sands of the soils, each of the minerals halloysite, montmorillonite, nontronite, and beidellite were identified in one sample or another, and although the contents of the minerals in the sands were low, montmorillonite in one silt sample amounted to over 50 per cent. Humbert and Marshall (1943) have also recorded the presence of clay minerals in a granite and a diabase soil from Missouri.

Carroll has examined the fine sand fractions of several of the soils considered in the present work and recorded the presence of clay minerals in all, in some instances in quite considerable amounts. In the fine sand fractions of soils 1048 and a second black earth in close proximity to 1216, she (Carroll 1931-2) has recorded a high percentage of "kaolinized material", and an examination of the coarse sands during the present investigation shows a content of about 75 per cent. of a soft greenish-white clayey material in each. The cation exchange capacities of these fine and coarse sands vary from 32 to 47 m.e.*

Clay minerals have also been identified in the fine and coarse sand fractions of the red-brown earth 1007 and the chestnut earth 1845, and according to Carroll and present examinations they would amount to from 10 to 20 per cent. in both fractions of the soils. The low exchange capacities of 6 to 10 m.e. are a reflection of the very much lower concentrations of clay minerals in these sands compared with the former.

Kaolinized material in quite appreciable amounts has been identified in the fine sand of a red loam from the locality from which 6508 was obtained, and in a smaller amount in soil 902. The exchange capacities of these two fractions are 27 and 7 m.e. respectively.

The fine sand fractions of the podsolized soil 6509 may be compared with those from the somewhat similar soils from Kuitpo in South Australia in which clay minerals have been recorded in amounts ranging from 30 to 60 per cent. of the light mineral fraction. The coarse sand fractions of this soil and 6508 above, after treatment with sodium sulphide to remove cementing iron oxides, yielded soft residues of an essentially clayey nature. The exchange capacities of the original coarse sand fractions of these two soils are 21 and 25 m.e. respectively.

* "m.e." = "milli-equivalents per 100 g.", unless otherwise stated.

The fine and coarse sand fractions of the podsolized soils 1375, 2187, 3401, and 3998, and the fine sand of the red-brown earth 3496, are highly siliceous and consist essentially of quartz grains admixed, in most cases, with feldspars showing evidence of alteration to clayey material. The coarse sand of 3496 is rich in ferruginous clayey material and has a capacity of 15 m.e.; the exchange capacities of the sands of the other soils are negligible. The fine sand of the podsolized soil 3948 is highly micaeous and has a small capacity of 3 m.e.

In view of the extremely high capacity of the soil 5386, developed on recent volcanic ash from New Guinea (Hosking 1938) the analyses of the fine sands from two profiles formed on similar materials in Java in the same volcanic belt are of particular interest. In these profiles the light mineral fractions of the fine sand fractions consist, in the deeper layers in particular, almost entirely of clay minerals. Carroll (1933-4) has examined this material in some detail and has separated it into kaolinite (about 20 per cent.) and "kaolinized material" (60 to 70 per cent). It is apparent that the former consists of both kaolinite ("hexagonal plates") and halloysite ("crystal aggregates of tiny fibres parallel to each other, similar to halloysite described by Ross and Kerr (1931, 1934)"); the kaolinized material is essentially amorphous but some "cryptocrystalline grains" have been related to nontronite. In other soils such kaolinized material has been related to the zeolites, and in view of the extremely high exchange of the permutite, an artificial zeolite, of 350 m.e. (Hosking 1948) the high concentration of this amorphous material is significant, not only in these soils but in most of the others already considered.

Since a definite upper limit of the order of only a few microns is imposed upon the size to which the individual clay mineral particles may attain, the presence of such large concentrations of the clay minerals in the coarser fractions of soils might be considered as casting some reflection on the efficiency of the dispersion methods used in mechanical analysis.

It must be realized, however, that various iron oxides, silica, and other materials deposited from the soil solution may act in soils as cementing agents, which result in an agglomeration of the finer particles into both porous and dense extremely stable particles. The preliminary weak acid treatment used in mechanical analysis is not designed to break down this cementation, but rather to dissolve out materials such as calcium carbonate and gypsum which, if allowed to remain, would result in subsequent flocculation of the colloids.

While kaolinite in the free state commonly occurs as particles with an equivalent diameter of about 1μ, the upper limit to the size of the individual crystals has been shown to vary from about 60 to 100μ (Hosking 1942; Ross and Kerr 1931). Similar upper limits have been observed for halloysite and amorphous clays related to the kaolin minerals (Ross and Kerr 1934). Coarser dense or massive aggregates not easily reduced by dispersion processes have also been identified.

In the case of the montmorillonite minerals, Humbert (1942) Marshall and his colleagues (1942), and Shaw and Humbert (1941) have shown that the individual crystals are fibrous, rod, or lath shaped,

and although of definitely colloidal dimensions have a very marked tendency to form equidimensional aggregates made up of a very large number of interlaced crystals. There may thus be built up large aggregates which are quite stable and resistant to strong dispersion. Too drastic a dispersion, however, must be avoided because of the fact that even slight intensification of the methods used in mechanical analysis might have appreciable effects in actually causing a breaking down of certain minerals, such for instance as the fibrous mineral attapulgite examined by Marshall and his colleagues (1942), into finer particles through the cleavage of the larger crystals.

It has been generally considered that the present standardized methods for mechanical analysis give a more or less complete dispersion of the soil into its ultimate particles. In actual practice it is now apparent that such dispersion has not been realized. However, more drastic treatments than at present adopted cannot be resorted to without breaking down certain of the more fragile primary crystals present in the soil. In consequence the present methods will probably have to stand and the fact be faced that in reality a compromise has been reached in the employment of such definitely arbitrary medium-drastic treatments.

The clay minerals present in the sands and silts separated according to the general standardized procedures may be considered as definite constituents thereof, and hence cation exchange reactions are just as characterisic a feature of the coarser fractions of soils as of the finer colloids or clays.

6. Contributions from the Various Fractions to the Cation Exchange Capacity of the Soil

On the basis of the mechanical analysis (colloid + coarse clay + silt + sand = 100 per cent.) given in Table 2, the contributions from the various fractions to the total cation exchange capacity of the mineral portion of each soil have been calculated. In Table 3 the exchange capacities of seven of the soils, obtained by adding these values, are compared with the capacities determined directly on the whole samples, and excellent agreement is found to occur; in no case is the difference between the two values greater than 1 m.e.

It is apparent that the contributions from the sands and silts is considerable in all cases. Of the soils with capacities of over 40 m.e., the silts and sands each contribute from 5 to 20 per cent. of the total. In soils 6508 and 6509 the coarse sands alone are responsible for 10 and 20 per cent. respectively of the exchange capacity of these soils, while the sands and silts combined account for about 40 per cent. in both cases. In soil 4373 the contribution from the silt is 25 per cent. and that from the sands and silt combined is no less than 45 per cent.

Among the soils with exchange capacities of less than 20 m.e., No. 6560 is outstanding, for in this soil the coarse sand is the principal contributor and alone accounts for no less than 67 per cent. of the exchange capacity. In soil 3948 the sand and silt fractions contribute 46 per cent. of the capacity. The sand and silt fractions of soils 3998 and 902 are responsible for about 12 per cent. of the total.

TABLE 2.—MECHANICAL ANALYSIS OF THE MINERAL PORTION OF THE SOILS

Fraction	1048	1216	1007	1845	4373	6508	6509	3496	1375	902	2187	3998	3948	6560
	%	%	%	%	%	%	%	%	%	%	%	%	%	%
Coarse sand ..	1·3	1·6	8·0	11·7	5·8	12·5	19·6	2·2	12·4	6·8	35·2	36·4	16·9	49·1
Fine sand ..	14·3	16·8	20·0	21·3	24·6	6·1	6·0	19·4	16·7	10·2	19·9	13·6	28·3	8·3
Very fine sand ..						10·3	9·1	27·7						9·5
Coarse silt ..	13·7	24·1	10·5	11·1	26·3	7·4	5·6	12·2	8·1	11·2	9·4	10·0	18·0	8·9
Fine silt ..						17·4	13·9	20·2						14·9
Coarse clay ..	5·8	7·3	3·0	3·3	3·4	13·9	5·0	5·2	4·0	10·5	3·1	3·0	4·0	3·0
Colloid	64·9	50 2	58·5	52·6	39·9	32·4	40·8	13·1	58·8	61·3	32·4	37·0	32·8	6·3

TABLE 3.—CATION EXCHANGE CAPACITIES OF THE SOILS AND THE CONTRIBUTIONS THERETO OF THE VARIOUS FRACTIONS

Fraction	1048	1216	1007	1845	4373	6508	6509	3496	1375	902	2187	3998	3948	6560
	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*	m.e. %*
Coarse sand ..	0·61	0·54	0·48	1·03	1·40	3·09	4·04	0·33	0·13	0·57	0·10	0·03	0·07	4·71
Fine sand ..	5·05	5·38	1·60	2·13	5·04	1·68	0·47	0·43	0·23	0·66	0·12	0·10	1·05	0·22
Very fine sand ..						2·85	0·76	0·83						0·36
Coarse silt ..	5·19	7·40	1·70	1·74	9·99	1·98	0·49	0·63	0·74	0·84	0·31	0·53	1·15	0·37
Fine silt ..						5·17	1·63	1·52						0·66
Coarse clay ..	4·58	4·61	1·06	1·07	1·66	4·89	0·89	0·90	0·80	2·00	0·23	0·26	0·34	0·18
Colloid	70·81	48·44	46·16	31·61	22·90	15·26	13·22	4·15	15·17	15·20	6·48	3·63	3·12	0·47
Total calculated ..	86·24	66·37	51·00	37·58	40·99	34·92	21·50	8·79	17·07	19·27	7·24	4·55	5·73	6·97
Total determined..	85·5	64·3	..	..	..	33·90	21·6	9·4	..	..	..	3·7	..	7·00
Whole soil ..	88·2	66·8	48·8	37·8	38·5	41·0	27·5	13·1	19·10	21·2	..	4·6	6·60	..
Soil organic carbon (%)	1·46	1·71	..	0·54	0·40	5·00	2·42	1·43	0·66	1·81	1·06	0·32	0·54	..

* m.e. per 100g. of soil.

TABLE 4.—CATION EXCHANGE CAPACITY OF ORGANIC MATTER

—		3496	1048	1375	6509	3998	1216	6508	902
		%	%	%	%	%	%	%	%
Organic carbon		1·43	1·46	0·66	2·42	0·32	1·71	5·0	1·81
Organic matter		2·5	2·5	1·14	4·2	0·55	3·0	8·6	3·1
		m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%	m.e.%
Exchange due to organic matter	(1)	4·9	4·9	..	6·9	0·9	4·4	10·1	..
	(2)	4·5	4·2	2·2	6·9	..	4·4	9·1	2·5
Exchange capacity per 100 g. organic carbon*	(1)	340	335	..	285	280	260	200	..
	(2)	310	290	335	285	..	260	180	140
Exchange capacity per 100 g. organic matter*	(1)	190	190	..	170	170	150	120	..
	(2)	180	170	190	170	..	150	110	80

* To the nearest 5 m.e.

(1) Exchange figures and calculations based on figures for the whole inorganic portion of soil.

(2) Exchange figures and calculations based on figures for the various fractions.

7. Cation Exchange Capacity of Soil Organic Matter

To calculate the cation exchange capacity of the soil organic matter, the contribution from this material to the total exchange capacity is divided by the proportion of the organic matter in the soil. This contribution is readily determined by subtracting from the exchange capacity of the soil, either the value obtained experimentally for the hydrogen peroxide treated sample (Hosking 1948) or the value obtained by adding the capacities of the various mineral fractions; these capacities it must be realized are not those given in Table 3, but the values recalculated on the basis of the mechanical analysis of the whole soil.

The relevant data for these calculations for certain of the soils, together with the exchange capacities calculated for both organic carbon and organic matter, are given in Table 4; quite close agreement between the results calculated according to either procedure is obtained.

Apart from a somewhat low value of 140 m.e. for soil 902, the exchange capacities per 100 g. of organic carbon vary from 200 to 340 m.e., with a mean of about 280 m.e. For organic matter the range in capacities, excluding once again the rather low value for soil 902, is from about 120 to 200 m.e., with a mean value of about 170 m.e.

Apart from certain of the lower values recorded by Piper (1938) which overlap the range of values obtained in the present work, these exchange capacities are very much lower than those previously obtained for organic matter as may be seen by an examination of Table 5.

TABLE 5.—COMPARISON OF THE CATION EXCHANGE CAPACITIES SO FAR RECORDED FOR ORGANIC MATTER

Author	Cation Exchange Capacity			
	m.e. per 100 g. Organic Carbon		m.e. per 100 g. Organic Matter	
	Mean	Range	Mean	Range
Hosking (present value)	280	200 to 340	170	120 to 190
Piper (1938)*	345	..	200	..
Piper (1938)†	380	220 to 610	220	130 to 350
Williams (1932)	455	..	260	..
Prescott and Hosking (1936)	512	..	300	..
Slater and Byers (1934)	623	..	360	..

* Soils from the Waite Institute. † Other red-brown earths from South Australia.

In previous calculations all contributions to the exchange capacity over and above that due to the clay content have been attributed to the organic matter. The magnitude of the error involved may now be gauged when it is realized that the total contributions from the silt and sand fractions to the exchange capacity may frequently amount to two or three times that from the organic matter itself. With the elimination now of the total contributions from the entire inorganic fraction of the soil the new value for the organic matter may therefore be considered as far more reliable than previous estimates.

The range in values obtained is indicative of the complexity of soil organic matter.

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Effect of Mechanical Composition and Nitrogen Content on the Water-stable Aggregation of Several Soils

*By G. B. Clarke, B.Sc.**

Summary

The effect of clay, silt, and fine sand content on water-stable aggregation has been studied in seven soils. A significant positive effect of clay and an insignificant effect of sand was found in all cases. Five of the seven soils examined showed a significant positive effect of silt. There was no significant difference between the effect of 5μ and 2μ clay on water-stable aggregation.

The statistical data indicated a curvilinear relation between water-stable aggregation and mechanical composition.

The effect of nitrogen content on water-stable aggregation was examined in two soils. No significant relation was found.

1. Introduction

In soil structure studies, the dependence of water-stable aggregation on the mechanical composition† has received the attention of several workers. Puri (9, 10) and his collaborators have shown that water-stable aggregation is dependent on the mechanical composition of the soil. In one of these papers (9) water-stable aggregation is expressed by the ratio of single values derived from the aggregate and mechanical analysis summation curves.

Many workers (3, 8, 12, 13) have recognized that clay is one of the major factors in aggregate stability. Rost and Rowles (11) and Baver (3) present data showing a positive correlation between water-stable aggregates and clay content. Rost and Rowles' correlation, however, appears to be forced. Clarke and Marshall (5) have used linear regression equations of aggregation on clay content, derived from samples taken from the A-horizon below the depth of tillage, to correct for mechanical composition differences within the depth of tillage. McHenry and Russell (7) have shown that percentage aggregates greater than 0·25 mm. can be expressed as a logarithmic function of clay content in an artificial bentonite-sand mixture.

The consideration of silt and sand as determining variables in relation to the water-stable aggregation of a soil has received little attention. Hide and Metzger (6) in a comparison between well-aggregated and poorly-aggregated fractions of the aggregate distribution, found that the well-aggregated material contained more clay and sand whereas the poorly-aggregated material contained more silt. The method of separation was recognized by them as a possible cause for the greater amount of sand in the well-aggregated fraction. The part played by silt and sand, however, has been considered mainly in studies of the mechanics of aggregation of artificial mixes. McHenry and Russell (7) found that the level of aggregation for aggregates larger than

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† Mechanical composition refers to the size-distribution of *primary* particles.

0·25 mm. is lowered by increasing the silt-sand ratio, and Sideri (13) has shown that, while a mixture of clay and " sand " (0·01–0·02 mm.) is structured below a certain " sand "-clay ratio, an increase in this ratio causes the structure to disappear.

The activity of 5μ clay compared with 1μ clay has been studied by Baver (3) who has shown that in several soils 5μ clay is more closely related to water-stable aggregation than 1μ clay.

Although the effects of several types of organic matter, of carbon content, and of the carbon-nitrogen ratio on water-stable aggregation have been examined elsewhere, the effect of nitrogen content has not been studied widely. Ackerman and Myers (1), working with two groups of claypan soils which had different agronomic histories, found a high positive correlation in one group and no correlation in the other. Hide and Metzger (6) showed a higher percentage of nitrogen to be present in a well-aggregated as compared with a poorly-aggregated fraction of the aggregate distribution.

In the present investigation, Aggregation (0·05 mm.) has been adopted to express water-stable aggregation. It is defined in an earlier paper (5) as the percentage of particles smaller than 50μ in aggregates larger than 50μ. This expression is similar to Baver's (4) state of aggregation and Alderfer and Merkle's (2) stability index, except that 50μ was chosen arbitrarily as the lower limit for water-stable aggregates.

2. Experimental

(*a*) *Description of Soils and Cultivation History*

The soils under investigation were collected from red-brown earth profiles at the Waite Institute and from two types occurring in the Murrumbidgee Irrigation Area, New South Wales. Soil Types A and B have been discussed in an earlier paper (5). In Type A the A-horizon is a silty loam, and in Type B it is a clay loam to loam.

Soil Type C occurs in the Murrumbidgee Irrigation Area, New South Wales. It is described elsewhere (14) as a member of the Hanwood Series and is typified by a loam surface soil over a light clay subsoil. Soils Ca and Cb are in an irrigated orchard and differ only in their cultivation history. Ca has been under clean culture and Cb under a cover of lucurne for the past twenty years. Prior to this their histories were similar.

Soil Type D also occurs in the Murrumbidgee Irrigation Area and conforms essentially to the type Beelbangera clay loam (14). A typical profile in the area sampled has a clay loam surface with a clay subsoil overlying clay with lime and limestone rubble. The structure of the soil in this area is considered to be poor, the lower half of the A-horizon (approximately 4–8 in.) being hard and compact with a high apparent density. Type D has been under irrigated orchard for a number of years.

The mechanical composition of all the soils is shown in Table 1. The number of samples taken from each soil is shown in Table 3.

TABLE 1.—MECHANICAL COMPOSITION OF SOILS SHOWING THE MEAN AND STANDARD DEVIATION OF THE PERCENTAGE OF EACH FRACTION

(Calculated on total oven-dry soil)

Soil	Sampling Depth (in.)	<2μ		2μ–20μ		20μ–50μ		>50μ	
		Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.
A	4 –8	19·8	2·96	32·5	6·01	25·3	1·24	22·4	6·96
B	4 –8	26·1	6·25	31·9	2·73	22·5	2·37	19·6	5·19
Ca_1	0 –4½	30·9	3·52	13·4	1·67	12·5	1·98	43·1	4·35
Ca_2	4½–8½	32·0	4·33	14·3	2·58	11·5	3·41	41·3	5·93
Cb_1	0 –4½	28·4	4·76	14·5	3·46	11·9	2·26	45·1	5·52
Cb_2	4½–8½	32·7	9·91	12·9	2·77	14·4	3·42	40·1	10·54
D	4 –8	41·8	7·96	16·5	2·92	14·3	3·00	27·4	5·77

(*b*) *Sampling*

In all soil types, samples were taken from the A-horizon only. In Types A and B, samples were collected below the depth of tillage (4 in.), to avoid the effect of differences in cultivation treatment of the surface soil. The sampling of these types has been discussed elsewhere (5).

In Type C, Ca_1 samples were taken from the surface soil (0–4½ in.) which lies in a zone of uniform tillage, and Ca_2 samples from below this depth (4½-8½ in.). Cb_1 and Cb_2 samples were taken from 0–4½ in. and 4½–8½ in. respectively. Type C samples were collected from an area of approximately 7 acres.

Type D samples were taken from the upper 4 in. of the compact horizon which commences at an average depth of 4 in. throughout the area sampled. This area was approximately 2·5 acres. The surface 0–4 in. was also sampled but the range of mechanical composition was too limited to enable a statistical analysis to be made for the present purposes.

(*c*) *Laboratory Methods*

The preliminary treatment of the samples and the methods of severe and gentle dispersion for determining the water-stable aggregation by hydrometer analyses have been fully discussed in an earlier paper (5).

A time of shaking of 20 minutes was adopted in the gentle dispersion procedure. It was found that in the soils studied the lower limit of 50μ did not differ greatly from the point of intersection of the aggregate and mechanical analysis distribution curves. Hence only a small percentage error was involved in expressing water-stable aggregation as Aggregation (0·05 mm.).

3. Water-stable Aggregates

Experimental data of the water-stable aggregation for all soils is shown in Table 2.

TABLE 2.—VALUES OF AGGREGATION (0·05 mm.) FOR SEVEN SOILS SHOWING THE MEAN AND STANDARD DEVIATION

(*Calculated on total oven-dry soil*)

Soil	Aggregation (0·05 mm.)	
	Mean	S.D.
A	20·1	5·45
B	23·1	8·12
Ca_1	15·7	4·44
Ca_2	19·4	4·80
Cb_1	20·7	3·83
Cb_2	22·3	6·95
D	28·1	6·09

(*a*) *Effect of 2μ Clay, 2μ–20μ Silt, and 20μ–50μ Sand*

The influence of all particles smaller than 50μ is implied in the term Aggregation (0·05 mm.). The expression of Aggregation (0·05 mm.) in terms of clay, silt, and lower fine sand, therefore, gives a logical representation of this measurement of water-stable aggregation.

A preliminary study of the aggregation and mechanical composition data showed that the range of Aggregation (0·05 mm.) could best be accounted for by a linear function of clay, silt, and lower fine sand, that is, expressed in the form

$$A = a + bx + cy + dz$$

where A is Aggregation (0·05 mm.), and the determining variables x, y, and z are respectively percentage of 2μ clay, 2μ–20μ silt, and 20μ–50μ sand. In the majority of soils studied x, y, and z were found to be statistically independent. Statistical data of the regressions of Aggregation (0·05 mm.) on clay, silt, and lower fine sand are shown in Table 3.

All the soils show a significant positive effect of clay and an insignificant effect of sand on the water-stable aggregation of these soils. Five of the soils, B, Ca_1, Cb_1, Cb_2, and D, show a significant positive effect of silt. The values of t show clay to exert the most consistent effect in determining the water-stable aggregation of these soils. The regression coefficients indicate that in six of the seven soils, unit change in clay content causes a greater change in water-stable aggregation than unit change in silt. The difference between

TABLE 3.—RELATION BETWEEN AGGREGATION (0·05 mm.) AND MECHANICAL COMPOSITION

(Regression coefficients of 2μ clay (b), 2μ–20μ silt (c), and 20μ–50μ sand (d) with values of t in brackets, multiple correlation coefficient (R), intercorrelation coefficients (r) and number of samples, are given for seven soils)

Soil	Regression Coefficients			R	Intercorrelation Coefficients			Number of Samples
	b	c	d		r_{xy}	r_{xz}	r_{yz}	
A ..	1·4204* (3·564)	0·0715 (0·241)	0·8283 (1·671)	0·866	0·855*	0·153	0·127	24
B ..	1·3934* (15·160)	0·6553* (3·610)	0·3404 (1·410)	0·965	−0·356	−0·590*	0·347	26
Ca_1 ..	1·0223* (7·363)	0·8181† (2·763)	−0·2333 (0·959)	0·935	0·226	−0·064	0·142	15
Ca_2 ..	0·7862* (4·140)	0·4614 (1·426)	0·5060 (2·068)	0·806	−0·068	0·023	−0·180	16
Cb_1 ..	0·7244* (7·837)	0·4219* (3·257)	0·3747 (1·888)	0·920	−0·108	−0·116	−0·207	16
Cb_2 ..	0·5840* (9·888)	0·7467* (3·706)	0·1417 (0·885)	0·967	0·403	−0·364	−0·206	16
D ..	0·9919* (3·540)	0·8282* (3·301)	0·9582 (1·281)	0·923	−0·007	−0·945*	−0·120	16

* Significant at the 1 per cent. level.
† Significant at the 2 per cent. level.

the regression coefficients on clay and on silt was significant for soil Cb_1 and highly significant for soils A and B. An overall χ^2–test (omitting the insignificant reversed difference for soil Cb_2) showed that on the whole the clay coefficient is greater than the silt coefficient ($\chi^2 = 78·44$ with 12 d.f.; the large value of χ^2 came mainly from the highly significant difference between the coefficients in soil A).

(b) Effect of 5μ Clay

The activity of 5μ clay and 2μ clay was compared by linear regression equations ($A = a + bx$)‡ of Aggregation (0·05 mm.) on 5μ and on 2μ clay. Statistical data of these linear regressions for all soils are shown in Table 4.

All the regression coefficients are significant at the 1 per cent. level. The data indicate that, on the whole, 5μ clay exerts a more consistent effect on the water-stable aggregation of these soils than 2μ clay, but is related to Aggregation (0·05 mm.) by a smaller regression coefficient. A test on the difference between the two regression coefficients showed soil A to be the only one in which the difference was significant ($P = 0·026$). An overall test (omitting the reversed differences found in soils B and Cb_1) showed no significant difference between

‡x represents 2μ or 5μ clay.

TABLE 4.—RELATION OF AGGREGATION (0·05 mm.) TO CONTENT OF 5μ AND 2μ CLAY

(*Regression coefficients of 5μ clay (b') and 2μ clay (b''), correlation coefficients (r') and (r''), and number of samples are given for seven soils*)

Soil	Regression Coefficients		Correlation Coefficients		Number of Samples
	b'	b''	r'	r''	
A	1·0734	1·5557	0·873	0·845	24
B	1·4932	1·1898	0·883	0·890	16
Ca_1	1·0814	1·1181	0·929	0·886	15
Ca_2	0·7440	0·7833	0·760	0·722	16
Cb_1	0·7038	0·6708	0·889	0·833	16
Cb_2	0·5760	0·6502	0·946	0·927	16
D	0·6422	0·6481	0·869	0·848	16

the 5μ and 2μ coefficients ($\chi^2 = 14·0011$ with 10 d.f.). In no case was the difference between the 5μ and 2μ correlation coefficients significant. An overall test (omitting the reversed differences in soil B) showed that the 5μ coefficients were not significantly greater than the 2μ coefficients ($\chi^2 = 12·9558$ with 12 d.f.). Increased sampling might show significant differences in the coefficients for these soils.

(*c*) *Effect of Nitrogen Content*

The effect of nitrogen content on water-stable aggregation has been studied in the samples collected from below the depth of tillage (4-8 in.) in soils A and B. Experimental data are shown in Table 5. The number of samples is shown in Table 6.

TABLE 5.—NITROGEN CONTENT FOR SOILS A AND B SHOWING THE MEAN, STANDARD DEVIATION AND RANGE

(*Percentage calculated on total oven-dry soil*)

Soil	Mean	S.D.	Range
A	0·112	0·0196	0·096
B	0·098	0·0175	0·067

Before examining the effect of nitrogen content, the influence of cultivation and mechanical composition in the zone below the depth of tillage is reviewed. In soil A there is an effect of the differential surface cultivation on the nitrogen content which is significant at the 5 per cent. level. This effect tends to improve the chances of a

correlation of Aggregation (0·05 mm.) with nitrogen content, since Aggregation (0·05 mm.) is affected by cultivation in a sympathetic manner. Soil B shows no significant effect of the differential surface cultivation on nitrogen content. A linear regression ($A = a + nN$) of Aggregation (0·05 mm.) on nitrogen content (N) has a regression coefficient significant at the 1 per cent. level. This significant coefficient is due, however, to the significant correlation of clay content with nitrogen content (cf. Table 6).

The deviations from the regression equations ($A = a + bx + cy$ in soil B and $A = a + bx$ in soil A) of Aggregation (0·05 mm.) on mechanical composition were plotted against nitrogen content. This showed a weak relation between Aggregation (0·05 mm.) and nitrogen content.

A summary of the regression equations (e.g. $A = a + bx + cy + nN$ as in soil B) of Aggregation (0·05 mm.) on the significant mechanical composition fractions (cf. Table 3) and nitrogen content is shown in Table 6. The ratio, Range : S.D., for the nitrogen content of samples of soil A and soil B is 4·90 and 3·83 respectively. The data indicate that in both soils, nitrogen content has no significant effect on water-stable aggregation.

This conclusion has been applied elsewhere (5) to a consideration of the causes of changes in structure accompanying cultivation of these soils. It does not conform to current viewpoints on the significance of organic matter in aggregate formation and may be a consequence of the low organic status of these soils. It is quite possible too that organic factors not represented by nitrogen content may have affected the water-stable aggregation of these soils.

TABLE 6.—RELATION OF AGGREGATION (0·05 mm.) TO MECHANICAL COMPOSITION AND NITROGEN CONTENT

(Regression coefficients of the significant mechanical composition fractions (b and c) and nitrogen (n) with values of t in brackets, multiple correlation coefficient (R), intercorrelation coefficients (r), and number of samples are given for soils A and B)

Soil	Regression Coefficients			R	Intercorrelation Coefficients			Number of Samples
	b	c	n		r_{yx}	r_{xN}	r_{yN}	
A ..	1·5491* (7·254)		17·2644 (0·536)	0·848	0·855*	0·058		24
B ..	1·3989* (11·983)	0·7941 (3·754)	−34·5044 (0·874)	0·963	−0·356	0·623*	0·144	26

* Significant at the 1 per cent. level.

4. The Constant a of the Regression Equations $A = a + bx + cy + dz$

While linear functions best fit the data of Aggregation (0·05 mm.) on mechanical composition in all the soils examined (cf. Table 3), the values for the constant a show that at low values of x, y, and z. these linear functions do not hold. Table 7 shows the value of this constant in each of the soils.

TABLE 7.—VALUES OF CONSTANT a IN THE REGRESSION EQUATIONS OF AGGREGATION (0·05 mm.) ON x, y AND z

Soil	A	B	Ca_1	Ca_2	Cb_1	Cb_2	D
Constant a ..	−23·81	−41·83	−23·93	−18·68	−10·45	−8·47	−40·73

When x, y, and z are zero, Aggregation (0·05 mm.) $=a$ which in all cases is negative. These negative values of a demonstrate a departure from the linear functions when the values of x, y, and z are low. Hence it is indicated that water-stable aggregation, expressed as Aggregation (0·05 mm.) must be related to clay, silt, and lower fine sand by a curvilinear function.

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An Investigation into the Inhomogeneity of Deformation in Wire Drawing and Rolling

By June Collins, B.Sc., and R. W. K. Honeycombe, M.Sc.**

Summary

A method involving the microscopic detection of the beginning of recrystallization in deformed metals is employed for investigating the inhomogeneity of familiar deformation processes.

It is shown that in drawn brass wires recrystallization may commence preferentially in two zones: firstly, at the surface of the wires as a result of additional deformation due to frictional forces and, secondly, in a zone intermediate between the surface and the centre. The surface layer of grains was not obtained after heavy deformations, nor was it obtained when new tungsten carbide dies were employed. However, in all cases, regardless of the nature of the dies and the extent of deformation, recrystallization commenced preferentially near the surface, the core being last to recrystallize, thus indicating that the deformation produced by wire drawing is inherently not homogeneous.

With rolled brass sheet, both the above types of inhomogeneity were observed at low deformations, but after heavier deformations the recrystallization commenced at random throughout the specimens, showing that the deformation was uniform on a macroscopic scale.

1. Introduction

The various methods available for the plastic deformation of metals vary both in the total deformation which they can give to a metal before failure and in the uniformity of their deformation. For example, it is well known that tensile deformation is very uniform as compared with the deformation produced in wire drawing or extrusion. Such differences are important both in practical applications where inhomogeneous deformation resulting in high residual stresses may be very undesirable, and in fundamental investigations where the lack of uniformity of deformation may lead to misinterpretation of results.

When a wire is drawn it is subjected to a tensile deformation, but in addition the outer layers of the wire shear in the opposite direction to the flow (5). Taylor and Quinney have shown that this shearing increases as the die angle becomes larger. It is also known that the friction between the metal surface and the die increases with increasing die angle (3). X-ray investigations have shown that the nature and extent of the preferred orientation varies from surface to centre of a drawn wire but this does not imply directly that the extent of deformation is greatly different.

Similar conditions exist in rolling, where the surface layers are not only compressed but, as in wire drawing, are sheared in the opposite direction to the rolling direction. X-ray diffraction investigations furthermore have shown that the surface layers of cold-rolled sheet are deformed less uniformly than the core.

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During an investigation of the recrystallization of α–β brass (2), it was noted that the deformed grains at or near the surface commenced to recrystallize before the grains in the centre of the wire, indicating that the deformation was not uniform. This behaviour was marked, and it was thought that it would be possible to investigate the inhomogeneity of deformation by this means. This paper describes experiments made on wires and on rolled strip, in which the inhomogeneity of deformation was investigated by the recrystallization method.

2. Experimental Method

(*a*) *Materials*

Brass was chosen as a suitable alloy for both the wire drawing and rolling experiments. The compositions were as follows:—

—	Brass Wire (0·192-in. Diameter)	Brass Sheet ($^5/_{16}$-in. Thickness)
	%	%
Copper	64·8	68·6
Lead	0·03	0·02
Tin	0·01	trace
Nickel	0·01	..
Iron	0·02	0·04
Zinc (balance)	35·1	31·3

The specimens were fully annealed prior to deformation.

(*b*) *Means of Deformation*

(i) *Wire drawing.*—Two series of experiments were carried out. In the first, 0·192-in. diameter wire was drawn by hand to various reductions in area through steel dies, whereas in the other series the wire was drawn mechanically through new tungsten carbide dies. The speed of drawing was 4 ft./minute. The various percentage reductions employed in the experiments were as follows:

Steel Dies		Tungsten Carbide Dies	
Diameter	Reduction	Diameter	Reduction
in.	%	in.	%
0·179	12·0	0·182	9·3
0·162	28·2	0·162	28·2
0·130	54·0	0·128	54·4
0·099	72·3	0·101	71·5
0·056	91·5	0·040	95·5

The angles of both sets of dies were very similar, the half-die angle in both cases being about 9°–10°. The carbide die surfaces were highly polished, whereas those of the steel dies were badly worn.

(ii) *Rolling.*—The rolling was carried out in a 6-in. two-high rolling mill, both rolls operating at the same surface speed. The rolls were lightly lubricated with oil. Specimens were rolled to the following reduction in area from 3/16-in. thick sheet.

Thickness	Reduction in Area
in.	%
0·163	12·5
0·139	25·7
0·094	50·0
0·052	76·0
0·020	90·0

(*c*) *Annealing of Specimens and Subsequent Examination*

The various specimens both rolled and drawn were annealed in the range in which recrystallization just commenced. This necessitated annealing a series of specimens over a range of temperature for each deformation, until the beginning of recrystallization was detected. After annealing, a thick electro-deposit of copper was plated on each specimen in order to preserve the surface during the polishing operation. The specimens were sectioned, then mounted in leucite, and subsequently electrolytically polished in aqueous orthophosphoric acid (1,000 g./litre H_3PO_4).

The specimens were etched in very dilute acid ferric chloride solution of the following composition:—

Ferric chloride, 10 g.
Hydrochloric acid, 1 ml.
Water, 200 ml.

This etchant proved the most satisfactory one for outlining the small recrystallized grains in a heavily deformed matrix of α-brass.

3. Experimental Results

(*a*) *Wire Drawing*

(i) *Steel dies.*—The first experiments were carried out with a series of steel dies, using a heavy grease as a lubricant. It was found that on annealing wires which had received only a small amount of deformation (12 per cent. reduction in area), the recrystallization commenced along the surface of the wire (Plate 2, Figs. 1–3). A well-defined layer of small recrystallized grains was obtained at the surface, while no recrystallization occurred elsewhere in the specimen (Plate 2, Fig. 3). Similar wires annealed at a higher temperature showed that the surface layer was more developed and recrystallization had commenced in many areas for some distance in from the surface. However, there was still no recrystallization at or near the centre of the wire.

A surface layer of recrystallized grains was also obtained on annealing wires drawn to 28 per cent. reduction in area (Plate 2, Figs. 4–6), however, in this case nucleation did not occur at the surface alone. Some centres of recrystallization simultaneously appeared in the body of the wire, although the centre of the wire was again slower to recrystallize.

After 54 per cent. reduction in area, the surface layer was not very distinct, and simultaneosly a greater number of centres of recrystallization had occurred throughout the specimen. Again, the recrystallization of the centre zone was less advanced than in the intermediate layers.

After still heavier deformation (72 and 91 per cent. reduction in area) the surface layer of recrystallized grains was no longer preferentially formed, but recrystallization occurred more readily near the surface than in the central portions of the wires. This behaviour was quite marked in the very heavily deformed wire (91 per cent. reduction in area) where the first nuclei occurred in a layer half-way between the surface and the centre of the wire (Plate 3, Figs. 1–3). At high temperatures, centres of recrystallization extended to the edge of the wire, leaving a central zone still unrecrystallized. Thus, even after 91 per cent. reduction in area, the central zone possessed different recrystallization characteristics to the rest of the wire.

The results obtained with the steel dies are summarized in Table 1.

TABLE 1.—RECRYSTALLIZATION OF BRASS WIRE DRAWN THROUGH STEEL DIES

Reduction in Area	Range of Temperatures Used in Annealing	Mode of Recrystallization
%	°C	
12	360–400	First occurs along well-defined continuous surface layer. Centre of wire last to recrystallize
28	300–320	First occurs along well-defined surface layer but also some centres in body of specimen
54	280–305	Small surface layer but large number of nuclei throughout specimen. Less in the centre of wire
72	245–260	No surface layer formed. Commences at random, with slightly less nuclei in centre
91	230–240	Commences in layer intermediate between centre and edge. Centre last to recrystallize

(ii) *Carbide dies.*—In order to determine whether this inhomogeneity of deformation, as indicated by the mode of recrystallization of the wires, was accentuated by the use of badly worn dies, some experiments were carried out with new carbide dies. These dies

were specially designed for the drawing of brass wire, and had a similar half-die angle (9°–10°) to the steel dies. Stearic acid was employed as a lubricant.

As in the previous experiments, a wide range of deformations (from 9 to 95 per cent. reduction in area) was employed. However, even at the lowest deformation, nucleation did not occur first at the surface of the wire—there was a complete absence of a surface layer. Plate 4, Figs. 1 and 2, shows the nucleation occurring in the mass of the wire rather than at the surface after 28 per cent. reduction. A similar deformation with the steel dies gave a well-defined surface layer (Plate 2, Figs. 4–6). However, at all deformations there was a central zone in which the recrystallization occurred at a later stage. Recrystallization was always more advanced in the outer zones of the specimens. This behaviour was particularly marked in the most heavily deformed wire (95 per cent. reduction, Plate 3, Figs. 3–6). In this wire the recrystallization commenced in a layer about half-way between the edge and the centre. At a later stage, the recrystallization extended towards the surface of the wire, leaving a central zone still unrecrystallized.

(iii) *Unlubricated die.*—A wire was given 28 per cent. reduction by drawing through unlubricated and degreased carbide dies as used in (ii). On annealing specimens it was found that there were indications of a surface layer of recrystallized grains, but again the main effect was greater recrystallization near the surface of the wire than in the central portion. Plate 4, Fig. 3, shows the very small surface layer obtained, while Plate 4, Fig. 4, shows the recrystallization commencing in the intermediate zone of the wire.

(*b*) *Rolling*

The 3/16-in. brass sheet after annealing was rolled to 12, 25, 50, 75, and 90 per cent. reduction in thickness. Specimens were annealed, then copper-plated and polished as in the case of the wires.

The sheet deformed by 12 per cent. reduction showed a distinct surface layer of recrystallized grains (Plate 4, Fig. 5). while the rest of the specimen was completely unrecrystallized. At a higher annealing temperature the recrystallization had occurred to a greater degree near the surface than in the centre of the specimen.

After 25 per cent. reduction in thickness, a small discontinuous layer of recrystallized grains occurred at the surface of the sheet, but nuclei were also present in the bulk of the alloy. Again the recrystallization was less advanced in the central zone.

No surface layer was obtained after 50 per cent. reduction in thickness, but recrystallization still occurred less readily in the central portion of the specimen. At higher deformations (75 and 90 per cent.) there was again no evidence of a surface layer, and the nuclei appeared completely at random throughout the specimen.

Table 2 summarizes the results obtained in the rolling experiments.

It should be mentioned that differences in behaviour were often detected between the top and under surfaces of the sheet. Recrystallization frequently occurred more readily on the undersurface than on the top surface.

TABLE 2.—RECRYSTALLIZATION OF BRASS SHEET AFTER COLD ROLLING

Reduction in Thickness	Range of Temperatures Used in Annealing	Mode of Recrystallization
%	°C	
12	330–340	Distinct surface layer formed—no nuclei in body of sheet. At higher temperatures layers near surface recrystallized more readily
25	340	Small discontinuous layer formed, also nuclei throughout specimen. Less advanced in central zones
50	295	No surface layer. Recrystallization occurred less readily in centre of sheet
75	250	No surface layer. Recrystallization uniform throughout sheet
90	240	No surface layer. Recrystallization uniform throughout sheet

4. Discussion and Conclusions

Since the tendency of a deformed metal to recrystallize depends on the deformation, it is concluded from the experiments that both the wire drawing and rolling processes result in marked inhomogeneity of deformation.

The wire drawing experiments show that the recrystallization of the specimens does not occur uniformly, but in preferred zones: (i) at the surface of the wire, and (ii) in a zone extending from the surface, but excluding the central part of the wire.

The formation of a surface layer indicates that the surface is relatively more heavily deformed than the bulk of the wire. This additional deformation is no doubt the result of friction between the die wall and the wire. The use of new lubricated carbide dies eliminated this effect completely, and in fact a surface layer could hardly be produced using the carbide dies in an unlubricated, grease-free condition. Using the steel dies, the surface layer was most pronounced at the low deformation where it might be expected that any difference in deformation between the surface and the core of the wire would be a maximum. The deformation due to frictional forces may be considered as a small contribution toward the general deformation, and it may become less significant as the general deformation is increased. Also, after large reductions in area, recovery may occur at the surface of the wire as a result of high local temperatures caused by friction. Both these factors mitigate against the formation of a surface layer.

The inhomogeneity which affects the bulk of the wire is clearly more significant as it occurs independently of whether the conditions of drawing are good or bad. That is, the inhomogeneity is present in wires drawn through well-lubricated, accurately-made carbide dies, as well as in wire drawn through crude worn steel dies. The zonal texture produced by wire drawing is well known (4). It has been clearly shown that the central portion of the wire has a much stronger fibre structure than the layer intermediate between the centre and the surface in which the flow is distinctly conical. Our experiments show that this central layer also tends to recrystallize at a later stage than the intermediate zones.

Taylor and Quinney's experiments indicate that shearing occurs in the vicinity of the surface, which may account for the greater inhomogeneity in the intermediate layer. However, Sachs has stated that this shearing decreases with increasing reduction and would be expected to be not very pronounced at heavy reductions. On the other hand, our experiments indicate that even after 95 per cent. reduction in area, recrystallization still commences preferentially in the intermediate zone.

These experiments have been limited to consideration of different deformations and dies. There are, however, other variables which have not been considered. For example, the actual die angle (which was constant in our experiments) if changed can have a marked effect on the distribution of the deformation in the wire. As the die angle increases the friction and the shearing of the surface layers increases, thus causing increased inhomogeneity of deformation. On the other hand, if the die angle is made very acute, then the conditions of deformation approximate to those present in wires stressed by tension, which is considered to be the method by which the most uniform deformation is obtained.

The rolling experiments indicate that, after small reductions, recrystallization commences at the surface, forming a distinct surface layer if the specimen is annealed at a low temperature. Again, this heavier deformation at the surface can be attributed to friction. Furthermore, the inhomogeneity persisted into the bulk of the metal, but was only detected up to deformations of 25 per cent. reduction in area. Specimens deformed to 50, 75, and 90 per cent. reduction in area appeared to recrystallize quite uniformly, indicating that at least at high deformations rolling produces a very homogeneous type of deformation.

These experiments have been concerned with the inhomogeneity of deformation over the whole cross section of the wire or sheet. It is known that plastic deformation is inhomogeneous even on a microscopic scale (1), namely, from grain to grain. This type of inhomogeneity was not important in our experiments as the material possessed a very fine grain size. If coarsely grained aggregates or even single crystals are subjected to similar treatment, then the inhomogeneity on the two scales are of importance.

5. Acknowledgments

The authors wish to express their thanks to Dr. W. Boas for his constant advice and encouragement, and Dr. S. H. Bastow for helpful criticism of the manuscript. They are indebted to Professor E. J. Hartung for laboratory facilities in the Chemistry Department, University of Melbourne.

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NOTES

New Electrical and Photometric Units

A decision of importance to all concerned with precise measurements of electrical or photometric quantities was made by the International Committee of Weights and Measures at its meeting held in Paris, in October 1946. It was then decided that new electrical and photometric units would be introduced on January 1, 1948. In accordance with this decision, the National Physical Laboratory, Teddington, England, and the National Bureau of Standards, Washington, U.S.A., have each issued statements concerning the new units.* The Australian electrical and photometric standards are based on those maintained at the National Physical Laboratory, and the practices that are being adopted by the National Physical Laboratory, as set out in the statement issued by that laboratory, are now being followed by the National Standards Laboratory of the Commonwealth in the case of the new electrical units and will very shortly be adopted in the case of the photometric units.

The official announcement issued by the Director of the National Physical Laboratory reads as follows:

Electrical Units.

In accordance with decisions taken by the International Committee of Weights and Measures at their meeting in Paris, 1946, the system of electrical units employed at the National Physical Laboratory will be changed on January 1, 1948. The units at present in use are the 'international' units, which are based on certain material standards. From January 1, 1948, the units employed will be those derived from the centimetre, gramme, and second, i.e. the so-called 'absolute' units. The effects of this change may be seen from the following table:

One international ohm	= 1·00049 'absolute' ohms
One international volt	= 1·00034 'absolute' volts
One international ampere	= 0·99985 'absolute' ampere
One international watt	= 1·00019 'absolute' watts
One international henry	= 1·00049 'absolute' henries
One international farad	= 0·99951 'absolute' farad

The factors given in this table apply to the mean international units; that is to say, to the mean values of the international units as realized at the various national laboratories.

After January 1, 1948, apparatus submitted to the Laboratory with a view to the issue of a National Physical Laboratory Certificate of Examination will be regarded as acceptable if its errors fall within the specified limits when the values are expressed in either international or absolute units, provided that it was manufactured before January 1, 1949, and is marked accordingly.

Apparatus manufactured after January 1, 1949, and any apparatus not marked with the date of manufacture, will be acceptable only if its errors fall within the specified limits when the values are expressed in absolute units.

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* For the official National Bureau of Standards announcement, see National Bureau of Standards Circular C459 " Announcement of Changes in Electrical and Photometric Units ", issued May 15, 1947.

The Unit of Light

Following the decision of the International Committee of Weights and Measures, the National Physical Laboratory will, on and after January 1, 1948, express all photometric values in terms of units based on the '*new candle*'. This unit of luminous intensity is of such a magnitude that the brightness of a full (or cavity) radiator (black body) at the temperature of solidification of platinum is 60 *new candles* per square centimetre. The *new lumen* is the luminous flux radiated within unit solid angle by a uniform source having a luminous intensity of one *new candle*. The new unit of illumination will be one *new lumen* per unit area One *new lumen* per square foot will also be called one new *foot-candle* and one *new lumen* per square metre one *new lux*. The new unit of brightness will be one *new candle* per unit area, and, alternatively, one *new foot-lambert*. The latter unit is defined as the brightness of a perfectly diffusing surface of 100 per cent. reflection factor when its illumination is one *new lumen* per square foot.

When differences of colour are involved in the determination of any magnitude in terms of the above units, the evaluation will be in accordance with that which would be obtained by an observer having the relative luminosity curve (curve connecting eye sensitivity with wavelength) adopted by the International Commission on Illumination in 1924 and later by the International Committee of Weights and Measures.

The difference between the new units and those in use hitherto are small. The present units, based on the 'international candle', were introduced on April 1, 1909, in Great Britain, France, and the United States of America and were adopted in 1921 by the International Commission on Illumination. Nevertheless, Germany and some Central European countries continued to use units based on the Hefner candle (about 0·9 international candle). The international candle was not based on any primary standard; it was derived from the units defined in terms of the old flame standards and was maintained by means of carbon and tungsten filament electric lamps. Further, the values of the unit at different colour temperatures did not exactly agree, when compared on the basis of the international relative luminosity curve adopted as 1924.

The magnitude of the new unit has been so chosen that it will introduce only very small changes (less than 0·5 per cent.) in the values of luminous intensity assigned to lamps operating at a colour temperature of about 2360°K. For lamps at considerably higher colour temperatures, e.g., ordinary gas-filled electric lamps at normal efficiencies, the values expressed in terms of the *new candle* will be several per cent. lower than those expressed in international candles. At the colour temperature of the primary standard (2046°K.), values in terms of the *new candle* are about 1·7 per cent. higher than those in terms of the international candle.

On account of this alteration in the basis on which sources of light of different colours are compared, it is impossible to give a factor for converting values in international candles to values in *new candles*.

The conditions for the issue of a Certificate for electrical apparatus submitted for test to the National Standards Laboratory will be those adopted by the National Physical Laboratory.

The National Standards Laboratory expects very shortly to complete the transfer of its photometric standards from the old to the new units and will then calibrate photometric standards submitted for test in terms of the new unit. In the meantime calibrations will be made, as in the past, in terms of the international candle and lumen.

Abstracts of Forthcoming Publications

THE AGE, RATE OF GROWTH, AND GENERAL LIFE-HISTORY OF THE AUSTRALIAN PILCHARD (*Sardinops neopilchardus*) IN NEW SOUTH WALES WATERS, by M. Blackburn, M.Sc. To be published as a Coun. Sci. Ind. Res. (Aust.) Bulletin.

The study deals with length frequency distributions, scale readings and maturity and general occurence data. The findings are compared with those for related species overseas.

The pilchards of New South Wales, which form part of the eastern Australian winter-spawning stock, attain average body (standard) lengths of about 91, 120, 144, and 162 mm. at the ages of 1½, 2, 3, and 4 years respectively. These sizes are equivalent to 4·2, 5·6, 6·6, and 7·5 inches total length. There are larger fish up to 196mm. body length (9·0 inches total), which include some at 5 and 6 years at least, but these are rare. In the second year there is fast growth in the spring and virtually none in the following winter, and probably the same applies in subsequent years.

As regards the general life-cycle, the larvae move inshore from the spawning areas and enter the inlets, where they metamorphose and stay until they are over a year old. The fish then form shoals and return to the sea, where they remain afterwards, though not venturing beyond the edge of the continental shelf. In this open-sea phase the pilchards are most conspicuous at the surface in the general winter season. These shoals are associated with spawning and the fish are mostly in poor condition, although many are still unripe at two years and some even at three. In the warmer parts of the year the adult fish are not often found at the surface, but those that do occur are fat. It is not thought that there are any large-scale migrations.

In respect both of growth-rate and general life-history these pilchards more closely resemble those of the Atlantic coast of south-western Europe than those of any other pilchard group (*Sardina* or *Sardinops*) yet studied. These others include those of California, Japan, and Britain which have a faster growth, and those of the Mediterranean which have a slower one.

Some special conclusions from the scale studies are as follows:

(i) The corresponding scale and fish-lengths are not in direct proportion, but over most of the range their increments are.

(ii) Great difficulty was encountered with omitted and secondary rings, and these cases mostly did not seem to be confined only to certain scales from the same fish. Secondary rings are held to be secondary in that they register events which affect only a minority of the population, not in that they are adventitious marks that appear on some scales only of the same individual.

(iii) The principal ("annual") rings are not all formed at the same season of the year. The first one appears in the spring and the subsequent ones in winter.

(iv) There is considerable variation in the lengths of fish at the time of formation of each age-ring, some fish having consistently greater lengths than others at all such periods

throughout life, and so seeming to have a faster growth. This is shown to be more apparent than real, because these are actually the older individuals of a year-brood, while there is no evidence of compensatory growth.

(v) Two main kinds of rings are formed upon the scales, of which one is generally associated with advancing sexual maturity. The other kind includes the first annual rings, the later ones where not obliterated by the subsequently formed "spawning marks" in mature fish, and the secondary rings. The probable circumstances of ring-formation are discussed.

Recent Publications of the Council

Since the last issue of this *Journal,* the following publications of the Council have been issued:

Bulletin No. 210.—"Preliminary Survey of the Natural Pastures of the New England District of New South Wales, and a General Discussion of Their Problems," by R. Roe, B.Sc. (Agric.).

This survey did not cover the whole of the New England district, so that no detailed information on the extent of the various pasture types was obtained. The district consists of tableland country at an elevation of 3,200 to 4,00 feet. The natural pastures are composed of predominantly summer-growing species, and production is at a standstill during the severe winter. Moist conditions also cause deterioration of the dry grass, and as a result grazing sheep tend to suffer from nutritional anaemia, weaners make little growth during their first year, and heavy losses of grown sheep often occur. The use of supplements such as cereal grains, concentrates, and mineral licks has been investigated and found to be ineffective or uneconomical. The use of sown pastures for winter grazing has been shown to provide a solution to the problem, but it has not been found possible to produce a good quality sown pasture on the granitic sandy soils. There is scope for intensive investigation of this problem. Some of the poor results obtained may have been due to the choice of unsuitable species for the environment. Mineral deficiencies probably contribute to the poverty of the soils. Where cultivation is practised, steps will have to be taken to prevent excessive soil erosion.

Bulletin No. 214.—"The Preparation and properties of Synthetic Cryolite," by P. Dixon, M.Sc., and T. R. Scott, M.Sc.

In view of the limited resources of natural cryolite, consideration is given to the methods of preparing synthetic cryolite. Methods for preparing suitable intermediate fluorine compounds from fluorspar and other sources are reviewed and a selection is given of the most promising methods for preparing cryolite from these soluble fluoride intermediates. Factors governing the choice of fluoride intermediates include insolubility in water, ease of preparation, and ease of purification. Methods for the production of hydrofluoric acid and ammonium fluoride, sufficiently free from combined silicon, are

described in detail, after which all but two methods for the preparation of cryolite are dismissed from further consideration, for reasons given.

Synthetic cryolite is found to possess a somewhat lower ratio of sodium fluoride to aluminium fluoride than the mineral. This deficiency of sodium fluoride is common to cryolite prepared by a wide variety of methods and is accompanied by the presence of water of constitution. A description is given of unsuccessful attempts to precipitate anhydrous cryolite. The purity of the product prepared from sodium aluminate solutions complies with the rigid specifications of the aluminium industry. The need for choosing suitable corrosion-resistant materials for use in the methods employing hydrofluoric acid is emphasized. Details are given of the analytical methods used in determining the composition of cryolite samples.

Bulletin No. 215.—" Studies in the Biology of the Skin and Fleece of Sheep. 4. The Hair Follicle Group and its Topographical Variations in the Skin of the Merino Foetus," by H. B. Carter, B.V.Sc., and Margaret H. Hardy, M.Sc.

Regional differences in the development and composition of the hair follicle group in the skin of twenty Merino foetuses are described. In the development of hair follicle groups the same sequence of events occurred in nearly every body region examined. The main differences between regions were due to the times of development and the relative sizes and numbers of primary and secondary follicles. These differences have been examined for the evidence they present of regional gradients in the skin. During the main period of hair follicle differentiation in the foetus each region examined on the trunk tended to be at a later stage of development than those posterior to it. This antero-posterior gradient was found on both dorsal and ventral parts of the trunk. No dorso-ventral gradient in time of development was detected, there being only minor differences between dorsal and ventral parts of the trunk and between the trunk and the limbs in this respect.

The findings are discussed in the light of the very limited information from other sources on hair follicle group development in the Merino and other breeds of sheep.

Bulletin No. 216.—" An Examination of the Peet-Grady Method for the Evaluation of Household Fly Sprays," by D. F. Waterhouse, M.Sc.

The Large Group Peet-Grady method is examined with particular reference to its suitability for the evaluation of household fly sprays. Conclusions are based on tests with pyrethrins, Thanite, and Lethane 384 standard. The future of the Peet-Grady method is discussed in the light, both of the results obtained and of the likelihood that DDT with its high toxicity and slow knockdown properties will be a component of all household fly sprays.; It is pointed out that the standard dosage of 12 ml. per 216 cu. ft. is far higher than normally used in actual practice and that this high dosage results in higher knockdown and mortality than when the spray is used in a room. On the other hand, a dosage of 2 ml. per 216 cu. ft. in the Peet-Grady chamber gave results similar to those obtained in room tests. Reduction in

exposure time from the standard 10 minutes to 1 minute produces a slight reduction in knockdown and mortality, but the reduction is not great. If the spray is introduced into the chamber while it is being aerated the reduction is greater, but conditions as severe as this are not likely to occur frequently in actual practice. A number of factors examined by other workers are brought together and discussed.

Bulletin No. 220.—"The Preparation and Use of Harvey's Reduced Strychnine Reagent in Oceanographical Chemistry," by D. Rochford, B.Sc.

A method involving the use of a continuous atmosphere of CO_2 is is given for the preparation of Harvey's reduced strychnine reagent. It is demonstrated by a statistical analysis of the calibration data of a reagent prepared in this manner that: (i) HCl addition to the sample improves its sensitivity; (ii) an optimum dilution of the stock reagent with sulphuric acid is characteristic of each reagent preparation; (iii) the optimum development time is below 1 hour; (iv) the working range of the reagent varies with the dilution; (v) two determinations of the "salt" error of the reagent are given; (vi) the "aging" effect varies from batch to batch, but in general an increase in sensitivity takes place with time; (vii) by the use of a 0·0025 per cent. safranin reference solution, the reagent is shown to have a much greater sensitivity than that of Cooper's, prepared according to Harvey's method; (viii) a method involving statistical procedures for the calibration of the reagent is presented.

The laboratory procedure for the use of this reagent is detailed, using a visual colorimeter and using a photoelectric absorptiometer.

Bulletin No. 222.—"The Chaetognatha of South-eastern Australia," by J. M. Thomson, M.Sc.

The identity of the species of Chaetognatha present in the plankton of south-eastern Australian waters is discussed. A classification of maturity stages in Chaetognaths is suggested. Degrees of protandry can be differentiated. Grouping of the epiplanktonic species into tropical, temperate, and cold-water forms is possible. Deep-water species are not so divisible. Tropical, temperate, and cold-water forms are distinguishable. Tropical species do not extend into Bass Strait. Only one tropical species shows a significant difference in the frequency of occurrence in neritic and oceanic waters. All but two of the non-tropical forms show significantly more frequent occurrence in the oceanic zone.

The possibility of using Chaetognatha as indicator organisms seems restricted to examining the degree of penetration by cold and warm water masses, northwards and southwards respectively, in the region between Bass Strait and Jervis Bay; and, possibly, to showing influxion of oceanic waters into the neritic zone.

Bulletin No. 225.—"Studies on the Control of Wheat Insects by Dusts. 1. Field Tests of Various Mineral Dusts against Grain Weevils," by F. J. Gay, B.Sc., D.I.C., F.N. Ratcliffe, B.A., and R. N. McCulloch, B.Sc., B.Sc.Agr. "2. Further Tests of Various Mineral Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C. "3. The Use of Dust Barriers for the

Control of Grain Insects," by F. J. Gay, B.Sc., D.I.C. "4. The Use of DDT-and 666-impregnated Dusts for the Control of Grain Pests," by F. J. Gay, B.Sc., D.I.C.

A number of mineral dusts, which had proved effective in laboratory experiments, were tested under conditions more closely approximating to those of normal storage. At Toowoomba, where conditions favoured the development of heavy infestations, magnesite and dolomite dusts maintained the wheat in reasonable condition for twelve months when mixed with the grain at the rate of less than 1 per cent. by weight, while untreated wheat was virtually destroyed in seven months. Near Sydney, where conditions were not so severe, the dusts maintained wheat in excellent condition for 18 months. Other experiments showed that a barrier of dust a foot wide might be useful in protecting isolated parcels of grain in warehouses and sheds from a wide range of stored products pests. Preliminary tests also showed the effectiveness of the insecticides DDT and 666 when incorporated in dusts. Pure 666 gave a good kill of the lesser grain borer, *Rhizopertha dominica,* even at the rate of 1 part in 15,000,000 of wheat, when applied mixed with magnesite.

Forthcoming Publications of the Council.

At the present time, the following future publications of the Council are in the press:

Bulletin No. 212.—"The Frictional Properties of Lead-base and Tin-base Bearing Alloys: The Role of the Matrix and the Hard Particles," by D. Tabor, Ph.D.

Bulletin No. 213.—"Laboratory and Field Tests of Mosquito Repellents," by R. N. McCulloch, B.Sc., B.Sc.Agr., and D. F. Waterhouse, M.Sc.

Bulletin No. 219.—"Spray Tests against Adult Mosquitoes. 1. Laboratory Spray Tests with Culicine (*Culex fatigans*) Adults," by D. F. Waterhouse, M.Sc. "2. Spray Tests with Anopheline (*Anopheles punctualatus farauti*) Adults," by D. F. Waterhouse, M.Sc., and D. O. Atherton, M.Sc.Agr.

Bulletin No. 221.—"Contributions to the study of the Cell Wall. 4. The Nature of Intercellular Adhesion in Delignified Tissue. 5. The Occurrence, Structure, and Properties of Certain Cell Wall Deformations," by A. B. Wardrop, M.Sc., and H. E. Dadswell, D.Sc.

Bulletin No. 223.—"Report of Marine Borer Survey in New Guinea Waters," by A. W. Shillinglaw, B.Sc., Dip.For., and D. D. Moore, B.Sc., A.S.T.C.

Bulletin No. 226.—"An Ecological Study of the Australian Plague Locust (*Chortoicetes terminifera* Walk.) in the Bogan-Macquarie Outbreak Area, N.S.W.," by L. R. Clark, M.Sc.

Bulletin No. 227.—"Studies on Perennial Veldt Grass (*Ehrharta calycina* Sm.)," by R. C. Rossiter, B.Sc.(Agric.).

Bulletin No. 228.—" Ecological Observations on the Small Plague Grasshopper, *Austroicetes cruciata* (Sauss.), in the Trangie District, Central Western New South Wales," by L. R. Clark, M.Sc.

Bulletin No. 229.—" Studies in Cement–Aggregate Reaction. I.—Australian Aggregates and Cements," by A. R. Alderman, D.Sc., Ph.D., A. J. Gaskin, M.Sc., R. H. Jones, B.Sc., and H. E. Vivian, B.Sc.Agr. " II.—The Effect of Alkali Movement in Hardened Mortar," by H. E. Vivian, B.Sc.Agr. " III.—The Effect of Void Space on Mortar Expansion," by H. E. Vivian, B.Sc.Agr. " IV.—The Effect of Expansion of the Tensile Strength of Mortar," by H. E. Vivian, B.Sc.Agr. " V.—The Effect of Void Space on the Tensile Strength Changes of Mortar," by H. E. Vivian, B.Sc.Agr. " VI.—The Effect of Carbon Dioxide," by A. J. Gaskin, M.Sc.

Bulletin No. 230.—" The Preparation and Properties of Aluminium Fluoride," by T. R. Scott, M.Sc.

Bulletin No. 231.—" Mint Weed, *Salvia reflexa* Hornem. Present Distribution and Status in Australia," by R. Roe, B.Sc.(Agric.), and N. H. Shaw, B.Agr.Sc.

Bulletin No. 232.—" Guide to the Medicinal and Poisonous Plants of Queensland," by L. J. Webb.

Bulletin No. 233.—" A Soil Survey of the Hundred of Seddon and Part of the Hundred of MacGillivray, Kangaroo Island, South Australia, including also Portions of the Hundreds of Cassini and Duncan," by K. H. Northcote, B.Agr.Sc., and B. M. Tucker, B.Sc.

Bulletin No. 234.—" Mineral Deficiency in Plants on the Soils of the Ninety-mile Plain in South Australia. Part 2," by D. S. Riceman, M.Sc., B.Agr.Sc.

Bulletin No. 236.—" Observations on the Plant Communities at Bundemar, Trangie District, New South Wales, in Relation to *Chortoicetes terminifera* (Walk.) and *Austroicetes cruciata* (Sauss.)," by L. R. Clark, M.Sc.

Bulletin No. 237.—" Heating in Stored Wheat. I. Respiration of Dry Grain, Insect Respiration, and Temperature and Moisture Effects ", by Joan Milthorpe, B.Sc., and R. N. Robertson, B.Sc., Ph.D. " II. Heat Production, Heat Conductivity, and Temperature Rise in Grain in the Presence and Absence of Insects," by R. N. Robertson, B.Sc., Ph.D. " III. Two Years, Temperature Records of Dry Grain in a Concrete Silo," by R. N. Robertson, B.Sc., Ph.D., and Joan Milthorpe, B.Sc.

Bulletin No. 238.—" Non-parasitic Disorders of Australian Apple Fruits," by W. M. Carne,

" Handbook of Australian Pelagic Tunicates," by Harold Thompson, M.A., D.Sc.

Experiments in the Use of DDT against the Pasture Cockchafer, *Aphodius howitti* Hope.
(See page 2.)

FIG. 1.—Apparatus used in the population counts of July and October 1946. The ¼-in. mesh sieve holds the soil excavated from the quadrat area; beside it is the ⅛-in. mesh sieve, and a steel 1-link quadrat. All are resting on a canvas square used to receive the earth from the first sieving.

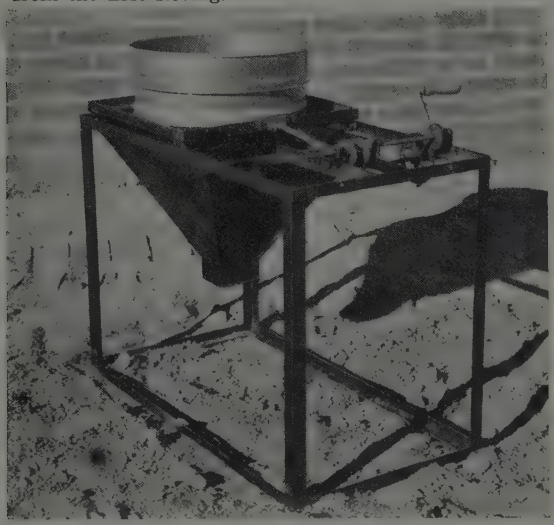


FIG. 2.—The sieving machine used in the population count of July 1947. The machine consists essentially of an angle-iron tray in which are mounted the two sieves, the ¼-in. one above. The tray, borne on four castors, is moved rapidly backwards and forwards by a crank geared to a handle. The sieved soil is collected in a bucket placed below the metal chute. A sheet metal cover is placed over the gears when the machine is in use.

PLATE 2

An Investigation into the Inhomogeneity of Deformation in Wire Drawing and Rolling. (See page 61.)

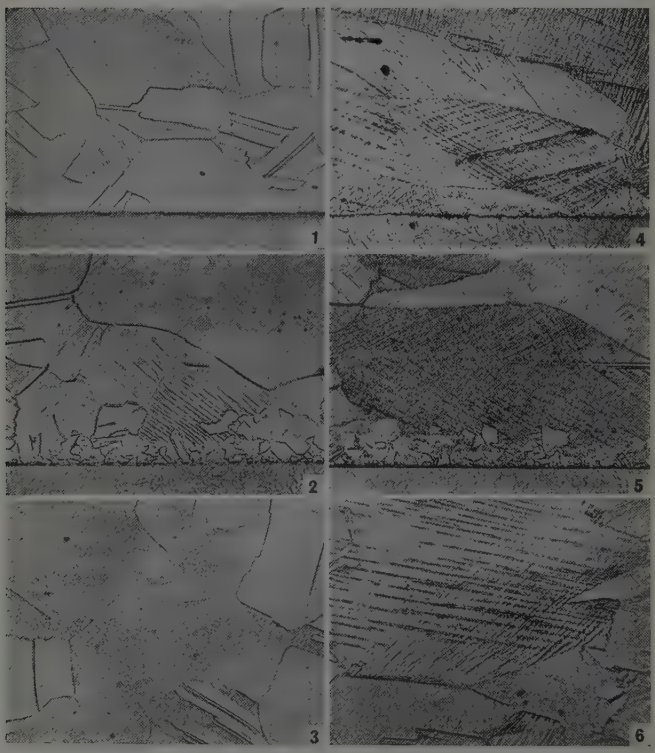


FIG. 1.—Area at surface of a wire after 12 per cent. reduction in area. As deformed—no recrystallization.

FIG. 2.—Similar area to that in Fig. 1 after annealing deformed wire 30 minutes at 360°C. Recrystallized layer is restricted to the vicinity of the surface.

FIG. 3.—Same specimen as in Fig. 2, but an area in the centre of the wire—no recrystallization.

FIG. 4.—Area at surface of a wire after 28 per cent. reduction in area. As deformed—no recrystallization.

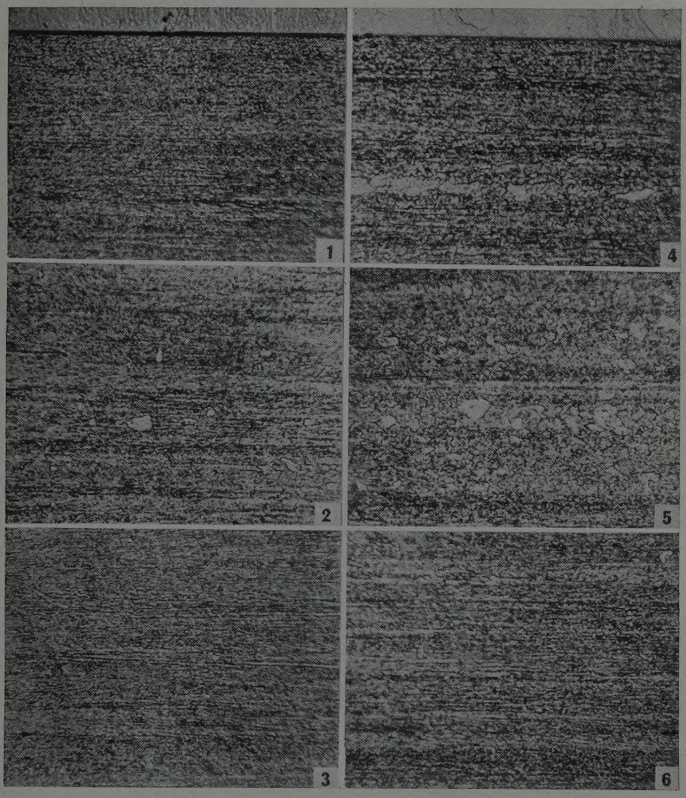
FIG. 5.—Similar area to that in Fig. 4 after annealing 30 minutes at 320°C. Recrystallization has commenced in a layer at the surface only.

FIG. 6.—Same specimen as in Fig. 5, but an area in the centre of the wire—no recrystallization.

(All photomicrographs × 400. The horizontal line near the bottom of Figs. 1, 2, 4, and 5 is the surface of the wire.)

PLATE 3

An Investigation into the Inhomogeneity of Deformation in Wire Drawing and Rolling. (See page 62.)



FIGS. 1–3.—Wire drawn through steel dies to 91 per cent. reduction in area then annealed 30 minutes at 240°C.

FIG. 1.—Area near surface of wire—no recrystallization.

FIG. 2.—Area half-way between surface and centre. Recrystallization has commenced.

FIG. 3.—Area at centre of wire—no recrystallization.

FIGS. 4–6.—Wire drawn through new carbide dies to 95 per cent. reduction in area then annealed 30 minutes at 230°C.

FIG. 4.—Area near surface. No recrystallization at surface, but has commenced at a short distance from the surface.

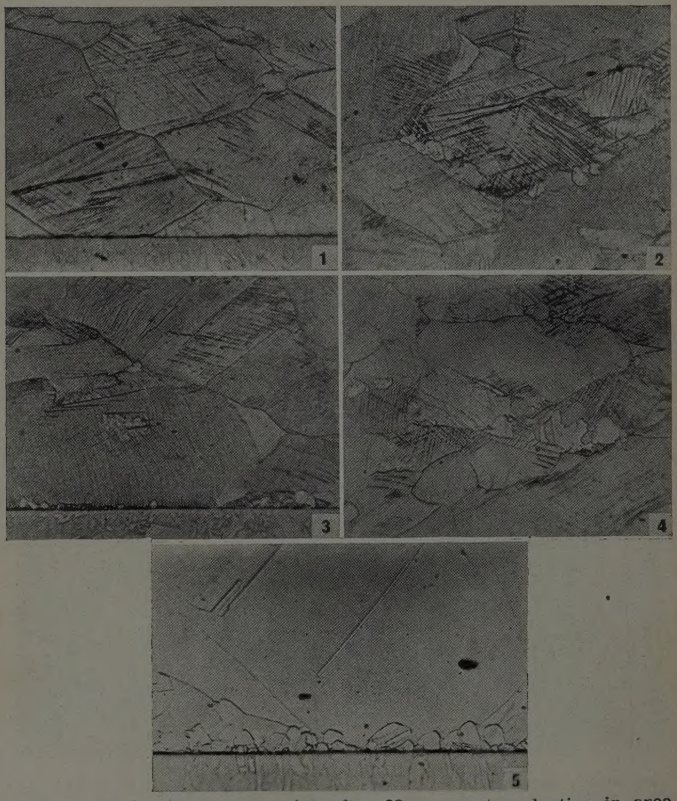
FIG. 5.—Area half-way between surface and centre—extensive recrystallization.

FIG. 6.—Area at centre of wire. Very slight evidence of recrystallization.

(All photomicrographs × 400. The horizontal line near the top of Figs. 1 and 4 is the surface of the wire.)

PLATE 4

An Investigation into the Inhomogeneity of Deformation in Wire Drawing and Rolling. (See page 63.)



FIGS. 1 and 2.—Areas on a wire after 28 per cent. reduction in area through carbide dies, followed by annealing 30 minutes at 310°C.

FIG. 1.—Area near surface showing no recrystallization.

FIG. 2.—Typical area showing recrystallization which has started in a zone between surface and centre.

FIGS. 3 and 4.—Areas on a wire after 28 per cent. reduction in area through unlubricated carbide dies.

FIG. 3.—Area near surface showing very weak development of a surface layer.

FIG. 4.—Area half-way between surface and centre where recrystallization has also started.

FIG. 5.—Area near the surface of a brass sheet rolled to 12 per cent. reduction in thickness then annealed 16 hours at 340°C. A. layer of recrystallized grains has formed at the surface.

(All photomicrographs × 400. The horizontal line near the bottom of Figs. 1, 3, and 5 is the surface of the wire.)

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